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#### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (46) 4753-4864 (2006)



#### Cover

See Robin D. Rogers et al., page 4767. Silver nanotubes,  $[Ag(pyrimidine)_2]_3[BF_4][NTf_2]_2$ were isolated from a complex mixture of ionic liquid, metal salt, and co-solvent, where the cosolvent provided metal ion solubility, the  $[BF_4]$  anions templated the coordination polymer, and the [NTf<sub>2</sub>]<sup>-</sup> anions insulated the nanotubes, decreasing electrostatic repulsions, and leaving very little void space in the structure. Image reproduced by permission of W. Matthew Reichert, John D. Holbrey, Kate B. Vigour, Tonya D. Morgan, Grant A. Broker and Robin D. Rogers from Chem. Commun., 2006, 4767.



#### Inside cover

See Stefanie Dehnen et al., page 4796. In search of watersoluble chalcogenometallate semiconductor compounds, a series of compounds with first discrete M/Sn/Te anions  $[M_4Sn_4Te_{17}]^{10-}$  or a polymeric derivative  $\{[Hg_4Sn_3Te_{13}]^6-\}_n$  of the ternary anionic structure all featuring optical absorption energies in the semiconductor range—have been synthesized by reacting K<sup>+</sup> (or Rb<sup>+</sup>) or Cs<sup>+</sup> salts of  $[SnTe_4]^{4-}$  with  $MCl_2$  (M = Mn, Zn, Cd, Hg) in H<sub>2</sub>O. Image reproduced by permission of Eugen Ruzin, Andreas Fuchs and Stefanie Dehnen from Chem. Commun., 2006, 4796.

#### **CHEMICAL TECHNOLOGY**

T45

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# **Chemical Technology**

December 2006/Volume 3/Issue 12

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#### **FEATURE ARTICLES**

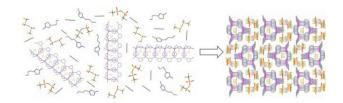
4767



Approaches to crystallization from ionic liquids: complex solvents-complex results, or, a strategy for controlled formation of new supramolecular architectures?

W. Matthew Reichert, John D. Holbrey, Kate B. Vigour, Tonya D. Morgan, Grant A. Broker and Robin D. Rogers\*

There are now more than 1200 papers a year describing research using ionic liquids as solvents, but comparatively little on their use in crystallization; we explore here why this is so and strategies for utilization of ionic liquids for crystallization and crystal engineering.



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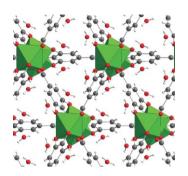
#### **FEATURE ARTICLES**

4780

#### Structural diversity and chemical trends in hybrid inorganic-organic framework materials

Anthony K. Cheetham,\* C. N. R. Rao\* and Russell K. Feller

We examine the emerging chemical and structural trends in the field of inorganic-organic framework structures and discuss some of the nascent applications of this vast and exciting class of materials.



#### **COMMUNICATIONS**

4796



Fine-tuning of optical properties with salts of discrete or polymeric, heterobimetallic telluride anions  $[M_4(\mu_4\text{-Te})(SnTe_4)_4]^{10^-}$  (M = Mn, Zn, Cd, Hg) and  ${}^3_{\circ}\{[Hg_4(\mu_4\text{-Te})(SnTe_4)_3]^{6^-}\}$ 

Eugen Ruzin, Andreas Fuchs and Stefanie Dehnen\*

Quaternary phases with fine-tuned optical absorption energies in the semiconductor range are obtained by reactions of alkali metal  $[SnTe_4]^{4-}$  salts with  $MCl_2$  (M = Mn, Zn, Cd, Hg) in  $H_2O$  or  $H_2O$ -MeOH.

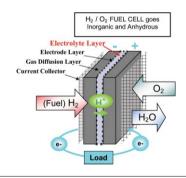


4799

#### Binary inorganic salt mixtures as high conductivity liquid electrolytes for >100 °C fuel cells

Jean-Philippe Belieres, Don Gervasio and C. Austen Angell\*

INORGANIC POWER: medium temperature (RT < T < 150 °C) H<sub>2</sub>–O<sub>2</sub> fuel cells perform well with new stable solvent-free inorganic ionic liquid electrolytes: low-current cell voltages can reach 1.2 V.



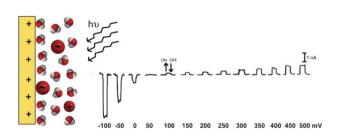
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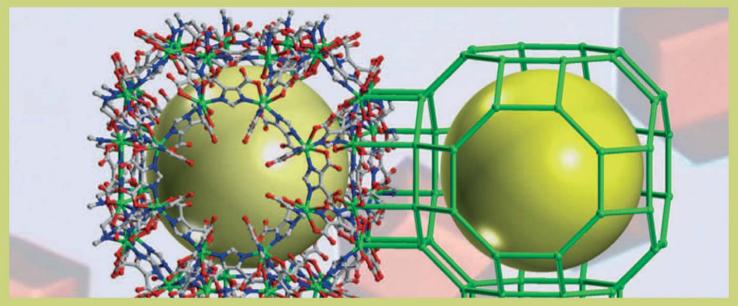


#### Investigation of laser induced photocurrent generation experiments

Himadri S. Mandal, Ian J. Burgess and Heinz-Bernhard Kraatz\*

Current signals produced by the laser-illumination of bare and non-chromophore containing peptide modified gold electrodes were investigated, and we suggest that these current signals which are due to the interfacial potential drop induced by laser heating, may have been mistakenly assigned to molecularbased photocurrents in several recent publications.





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# Professor T. Don Tilley US Associate Editor for inorganic, organometallic and materials chemistry

Don Tilley is Professor of Chemistry at the University of California, Berkeley. His research involves synthetic, structural and reactivity studies in organometallic systems. Metal-mediated routes to new polymers, and molecular approaches to the designed construction of advanced solid state materials and heterogeneous catalysts are also being developed.

Happy to receive papers on important developments in inorganic, organometallic and materials chemistry, Professor Tilley can be contacted via **chemcomm@berkeley.edu** 



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4805

#### A highly water-soluble C<sub>60</sub>-NVP copolymer: a potential material for photodynamic therapy

Yuko Iwamoto and Yoko Yamakoshi\*

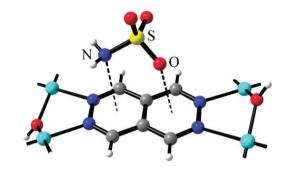
A water-soluble  $C_{60}$  polymer with the highest solubility (7.8 mM estimated as C<sub>60</sub> monomer) was prepared by a copolymerization with *N*-vinylpyrrolidone giving 30–50 kDa polymers with possible applications in photodynamic therapy.

4808

#### Metal-organic frameworks exhibiting strong anion- $\pi$ interactions

Il'ya A. Gural'skiy, Pavlo V. Solntsev, Harald Krautscheid and Konstantin V. Domasevitch\*

Pyridazino[4,5-d]pyridazine is a unique bicyclic  $\pi$ , $\pi$ -acidic system that retains efficient N-donor properties towards transition metal ions. Coordination polymers typically support strong anion- $\pi$  interactions and reveal an unprecedented double anion- $\pi$ , $\pi$  binding.

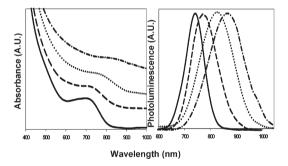


4811

#### $InAs_xSb_{1-x}$ alloy nanocrystals for use in the near infrared

Sang-Wook Kim,\* Sujith S. and Bun Yeoul Lee

 $InAs_xSb_{1-x}$  alloy nanocrystals for the near-infrared, which have quite a monodisperse crystalline structure of 2.5–3.0 nm and are of a zinc blend structure, are developed.



4814

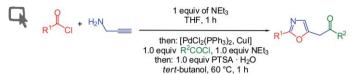
#### Microwave enhanced palladium catalysed coupling reactions: A diversity-oriented synthesis approach to functionalised flavones

Richard J. Fitzmaurice, Zac C. Etheridge, Emelie Jumel, Derek N. Woolfson and Stephen Caddick\*

The utility of diversity-oriented synthesis using palladium catalysed protocols with microwave heating is highlighted by application to functionalised flavones.

$$X = H, Br, OTf$$
 $X = H, Ar, NHnC_6H_{13}$ 

4817



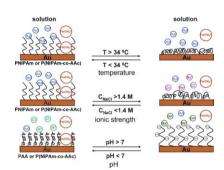
#### A new consecutive three-component oxazole synthesis by an amidation-coupling-cycloisomerization (ACCI) sequence

Eugen Merkul and Thomas J. J. Müller\*

Amidation, alkynylation and cycloisomerization in a one-pot sequence: A novel consecutive three-component synthesis of 1-(hetero)aryl-2-(2-(hetero)aryl-oxazol-5-yl) ethanones starting from propargyl amine and acid chlorides, both for amidation and cross-coupling, is based upon an amidation-coupling-cycloisomerization (ACCI) sequence.

4820





Temperature, ionic strength and pH induced electrochemical switching of smart polymer interfaces

Jianhua Zhou, Geng Wang, Jianqiang Hu, Xianbo Lu and Jinghong Li\*

A reversible electrochemical switching has been displayed at smart polymer brush interfaces, which was responsive to temperature, ionic strength and pH stimuli, independently or simultaneously.

4823

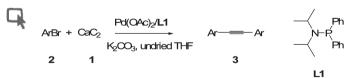
$$O \leftarrow \underbrace{ F_{20} \text{TPPFe(III)CI} \atop t\text{-BuOOH} \atop \text{CH}_2 \text{CI}_2\text{-MeOH} } \underbrace{ F_{20} \text{TPPFe(III)CI} \atop t\text{-BuOOH} \atop \text{CH}_3 \text{CN-H}_2 \text{O} }$$

Cytochrome P-450 model compound catalyzed selective hydroxylation of C-H bonds: Dramatic solvent effect

Arunava Agarwala and Debkumar Bandyopadhyay\*

Selective hydroxylation of cyclohexane and cyclohexene by t-BuOOH in presence of  $F_{20}$ TPPFe(III)Cl as the catalyst has been achieved at room temperature in high yields.

4826



The use of calcium carbide in one-pot synthesis of symmetric diaryl ethynes

Weiwei Zhang, Huayue Wu, Zhiqing Liu, Ping Zhong, Lin Zhang, Xiaobo Huang and Jiang Cheng\*

Symmetric diaryl ethynes could be prepared by the combination of aryl bromides,  $CaC_2$ ,  $Pd(OAc)_2$ , L1 and  $K_2CO_3$  in undried THF in one-pot.

4829

Water-soluble diruthenium complexes bearing acetate and carbonate bridges: highly efficient catalysts for aerobic oxidation of alcohols in water

Naruyoshi Komiya,\* Takahiro Nakae, Hideomi Sato and Takeshi Naota\*

The aerobic oxidation of alcohols in water can be performed efficiently in the presence of a catalytic amount of the water-soluble diruthenium complex  $Ru_2(\mu\text{-OAc})_3(\mu\text{-CO}_3)$  under an atmospheric pressure (1 atm) of  $O_2$ .

4832

Watson-Crick pairing of nucleobases functionalized with open-shell molecular entities in crystalline solids

Tomoaki Ise, Daisuke Shiomi,\* Kazunobu Sato and Takeji Takui\*

A Watson–Crick type molecular complex of adenine and thymine bases substituted with the stable radical of nitronylnitroxide has been synthesized, which forms a double-chain spin system in the crystal of the complex.

4835

A flow reactor process for the synthesis of peptides utilizing immobilized reagents, scavengers and catch and release protocols

Ian R. Baxendale, Steven V. Ley,\* Christopher D. Smith and Geoffrey K. Tranmer

A general flow process for the multi-step assembly of peptides has been developed to construct Boc, Cbz and Fmoc *N*-protected systems in excellent yields and purities.

4838

A new asymmetric organocatalytic nitrocyclopropanation reaction

Henriette M. Hansen, Deborah A. Longbottom and Steven V. Lev\*

Using 5-(pyrrolidin-2-yl)-1*H*-tetrazole as an organic catalyst, the nitrocyclopropanation of 2-cyclohexen-1-one has been achieved, proceeding in high yield and with good enantioselective control.

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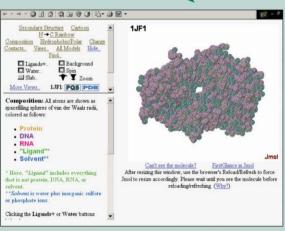
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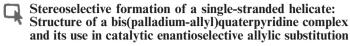


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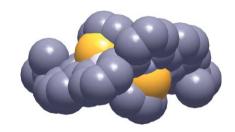
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4841



Hoi-Lun Kwong,\* Ho-Lun Yeung, Wing-Sze Lee and Wing-Tak Wong

Chiral  $C_2$ -symmetric quaterpyridine reacts with  $[Pd(\eta_3-C_3H_5)Cl]_2$  stereoselectivity to form a single-stranded helical binuclear palladium complex which catalyze asymmetric allylic substitution.



4844

Facile preparation of fluorovinylene aryl ether telechelic polymers with dual functionality for thermal chain extension and tandem crosslinking

Scott T. Iacono, Stephen M. Budy, Dirk Ewald and Dennis W. Smith, Jr.\*

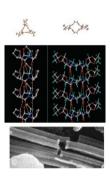
The facile preparation of new fluorovinylene aromatic ether polymers *via* step-growth addition polymerization of commercial bis(trifluorovinyl) aromatic ethers and bisphenols is described. The resulting polymers possess dual fluoroolefin functionality, allowing tandem thermally-initiated chain extension and crosslinking.

4847

Self-assembly of cyclic homo- and hetero-β-peptides with cis- furanoid sugar amino acid and β-hGly as building blocks

Bulusu Jagannadh,\* Marepally Srinivasa Reddy, Chennamaneni Lohitha Rao, Anabathula Prabhakar, Bharatam Jagadeesh\* and Srivari Chandrasekhar\*

A new class of peptide nanotubes, self-assembled from cyclic homo- and hetero- $\beta$ -peptides based on *cis*-furanoid sugar amino acid and  $\beta$ -hGly residues are described and represent the expansion of the conformational pool of *cis*  $\beta$ -sugar amino acids in the design of peptide nanotubes.



4850

Phosphazene base-promoted functionalization of aryltrimethylsilanes

Koichi Suzawa, Masahiro Ueno, Andrew E. H. Wheatley and Yoshinori Kondo\*

Selective functionalizations of arylsilanes were accomplished using *t*-Bu-P4 base catalyst.

$$X = \frac{\text{SiMe}_3}{t\text{-Bu-P4 base}}$$

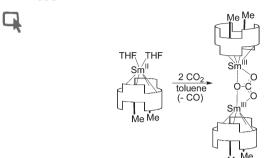
RCHO

 $t\text{-Bu-P4 base}$ 

(catalyst)

room temp.

4853

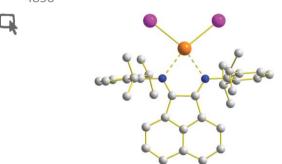


Reductive disproportionation of carbon dioxide by a Sm(II) complex: Unprecedented f-block element reactivity giving a carbonate complex

Noel W. Davies, Alistair S. P. Frey, Michael G. Gardiner\* and Jun Wang

Carbon dioxide reduction by a Sm(II) complex featuring a dimetallated *N*-methyl substituted porphyrinogen gives a bimetallic Sm(III) carbonate complex and carbon monoxide, representing the first example of reductive disproportionation by an f-block element.

4856

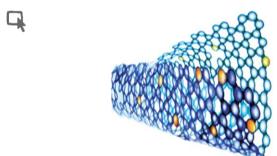


Direct reactions of tellurium tetrahalides with chelating nitrogen ligands. Trapping of  $TeI_2$  by a 1,2-bis(arylimino)acenaphthene (aryl-BIAN) ligand and C-H activation of an  $\alpha,\alpha'$ -diiminopyridine (DIMPY) ligand

Gregor Reeske and Alan H. Cowley\*

The reaction of TeI<sub>4</sub> with the dpp-BIAN ligand results in the Te(II) complex (dpp-BIAN)TeI<sub>2</sub> while treatment of TeCl<sub>4</sub> with the dpp-DIMPY ligand causes C–H activation of an imino methyl group.

4859



## Nitrogen-containing carbon nanotubes as solid base catalysts

Stefan van Dommele, Krijn P. de Jong and Johannes H. Bitter\*

Re-usable solid base catalysts were prepared in the form of nitrogen-containing carbon nanotubes (NCNT). The materials are conveniently prepared by catalytic chemical vapour depositions. The activity for a Knoevenagel condensation is related to the amount of pyridinic nitrogen incorporated in the NCNT structure and could be tuned by the synthesis parameters of the NCNT.

#### **ADDITIONS AND CORRECTIONS**

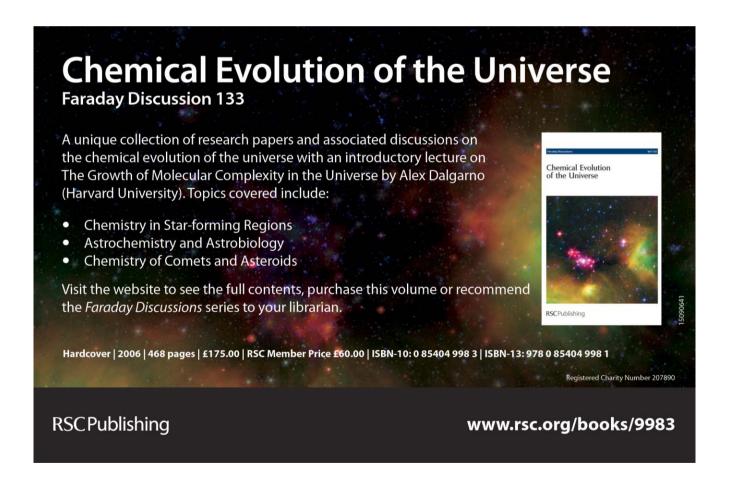
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In situ investigation of the oxidative addition in homogeneous Pd catalysts by synchronised time resolved **UV-Vis/EXAFS** 

Gemma Guilera, Mark A. Newton, Charlene Polli, Sakura Pascarelli, Meritxell Guinó and King Kuok (Mimi) Hii

A ratiometric and non-enzymatic luminescence assay for uric acid: differential quenching of lanthanide excited states by anti-oxidants

Robert A. Poole, Filip Kielar, Siobhan L. Richardson, Philip A. Stenson and David Parker



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