

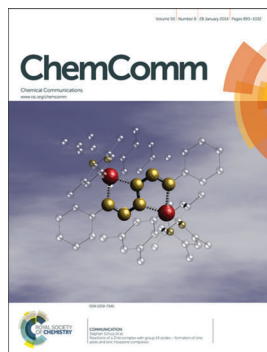
## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS 50(8) 893-1032 (2014)



### Cover

See Fernando Benito-Lopez, Dermot Diamond *et al.*, pp. 924-926. Image reproduced by permission of Larisa Florea from *Chem. Commun.*, 2014, 50, 924.



### Inside cover

See Stephan Schulz *et al.*, pp. 927-929. Image reproduced by permission of Stephan Schulz from *Chem. Commun.*, 2014, 50, 927.

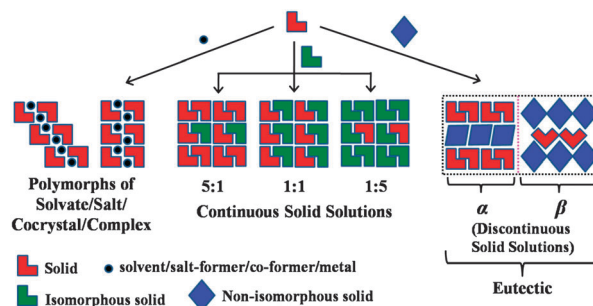
## FEATURE ARTICLE

906

### Eutectics as improved pharmaceutical materials: design, properties and characterization

Suryanarayan Cherukuvada and Ashwini Nangia\*

The combination of isomorphous solids gives rise to continuous solid solutions and solids in which the adhesive interactions outweigh the cohesive ones lead to cocrystals. With weak adhesive, strong cohesive and a geometric misfit, the product is eutectic.



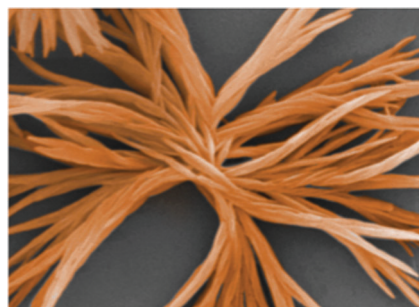
## COMMUNICATIONS

924

### Self-assembled solvato-morphologically controlled photochromic crystals

Larisa Florea, Silvia Scarmagnani, Fernando Benito-Lopez\* and Dermot Diamond\*

Self-assembled spiropyran microstructures exhibiting reversible photochromism upon light irradiation in the solid state are described for the first time. Guided assembling dimensions and aggregation of the microstructures at the liquid/air interface was also demonstrated.



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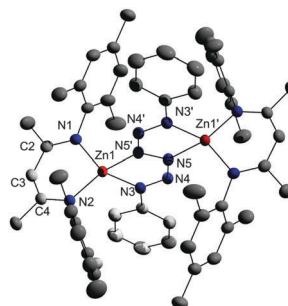


927

### Reactions of a Zn(II) complex with group 14 azides – formation of zinc azide and zinc hexazene complexes

S. Gondzik, S. Schulz,\* D. Bläser, C. Wölper, R. Haack and G. Jansen

Reactions of  $\text{Mesnacnac}_2\text{Zn}_2$  with group 14 azides either yield the zinc azide  $[\text{LZn}(\mu\text{-N}_3)]_2$  (**2**) or zinc hexazene complexes  $\text{L}_2\text{Zn}_2(\mu\text{-1,6-R}_2\text{-N}_6)$  ( $\text{R} = \text{Ph}$  **3**,  $\text{Dipp} = 2,6\text{-i-Pr}_2\text{C}_6\text{H}_3$  **4**).

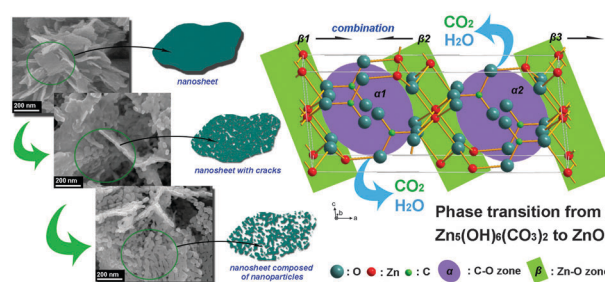


930

### Synthesis of hierarchically structured ZnO nanomaterials via a supercritical assisted solvothermal process

Meng Wang, Bin Zhao,\* Shaohong Xu, Lin Lin, Sijun Liu and Dannong He\*

Hierarchically structured ZnO nanomaterials with flower-sheet-particle morphologies were synthesized via a supercritical assisted solvothermal process free from any other auxiliary chemicals.

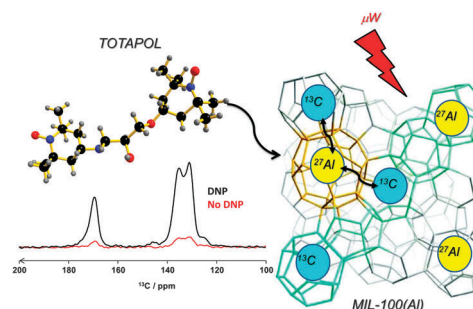


933

### Probing $^{27}\text{Al}$ – $^{13}\text{C}$ proximities in metal–organic frameworks using dynamic nuclear polarization enhanced NMR spectroscopy

Frédérique Pourpoint,\* Aany Sofia Lilly Thankamony, Christophe Volklinger, Thierry Loiseau, Julien Trébosc, Fabien Aussenac, Diego Carnevale, Geoffrey Bodenhausen, Hervé Vezin, Olivier Lafon\* and Jean-Paul Amoureux

We show how  $^{27}\text{Al}$ – $^{13}\text{C}$  proximities in the metal–organic frameworks MIL-100(Al) can be probed using advanced  $^{27}\text{Al}$ – $^{13}\text{C}$  NMR methods boosted by DNP.

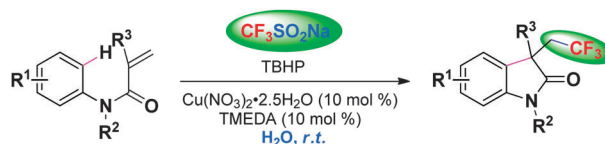


936

### Copper-catalyzed trifluoromethylation of *N*-arylacrylamides "on water" at room temperature

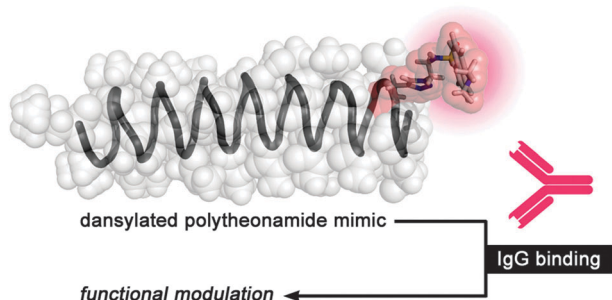
Fang Yang, Piyatida Klumphu, Yong-Min Liang\* and Bruce H. Lipshutz\*

A copper-catalyzed trifluoromethylations of arylacrylamides "on water" is reported using the stable and inexpensive Langlois reagent ( $\text{CF}_3\text{SO}_2\text{Na}$ ) en route to oxindoles.



## COMMUNICATIONS

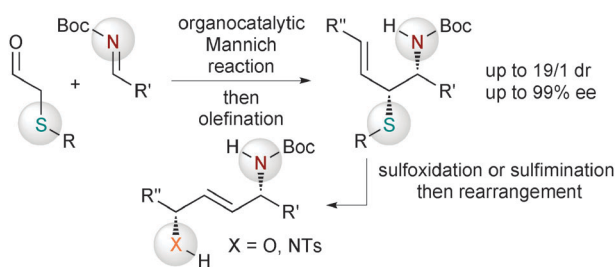
939

**Antibody-mediated functional control of a dansylated polytheonamide mimic**

Hiroaki Itoh and Masayuki Inoue\*

The function of a dansylated polytheonamide mimic, an artificial ion channel-forming cytotoxic peptide, was controlled by its specific interaction with an anti-dansyl antibody (IgG).

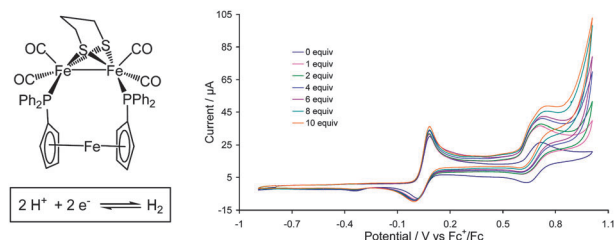
942

**Remote chirality control based on the organocatalytic asymmetric Mannich reaction of  $\alpha$ -thio acetaldehydes**

Taichi Kano, Ryu Sakamoto and Keiji Maruoka\*

Remote chirality control leading to 1,4-difunctionalized compounds such as 1,4-amino alcohols and 1,4-diamines was achieved by both *syn*- and *anti*-selective asymmetric Mannich reactions of  $\alpha$ -thio acetaldehydes, the subsequent olefination and the stereospecific 2,3-sigmatropic rearrangement.

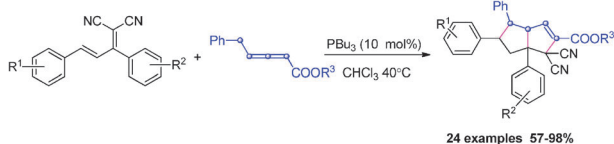
945

**Hydrogenase biomimetics:  $\text{Fe}_2(\text{CO})_4(\mu\text{-dppf})(\mu\text{-pdt})$  (dppf = 1,1'-bis(diphenylphosphino)ferrocene) both a proton-reduction and hydrogen oxidation catalyst**

Shishir Ghosh, Graeme Hogarth,\* Nathan Hollingsworth, Katherine B. Holt,\* Shariff E. Kabir and Ben E. Sanchez

The diiron complex  $\text{Fe}_2(\text{CO})_4(\mu\text{-dppf})(\mu\text{-pdt})$  is an active catalyst for both the reduction of protons to give hydrogen and also the reverse oxidation of hydrogen and thus mimics hydrogenases which are able to catalyse both reactions.

948

**Phosphine-catalyzed domino reaction: a novel sequential [2+3] and [3+2] annulation reaction of  $\gamma$ -substituent allenolates to construct bicyclic[3, 3, 0]octene derivatives**

Erqing Li and You Huang\*

A novel and efficient phosphine-catalyzed domino sequential annulation reaction of  $\gamma$ -substituent allenolates to construct bicyclic[3,3,0]octene derivatives is developed.



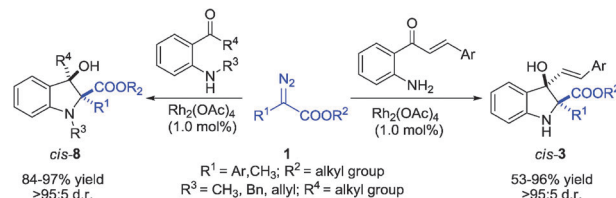
## COMMUNICATIONS

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### Highly diastereoselective synthesis of 3-hydroxy-2,2,3-trisubstituted indolines *via* intramolecular trapping of ammonium ylides with ketones

Changcheng Jing, Dong Xing and Wenhao Hu\*

A  $\text{Rh}_2(\text{OAc})_4$ -catalyzed diazo decomposition reaction of diazo esters with 2-aminophenyl ketones is reported. A series of 3-hydroxy-2,2,3-trisubstituted indolines are produced in good yields with excellent diastereoselectivities *via* an intramolecular aldol-type trapping of ammonium ylides with ketone units.

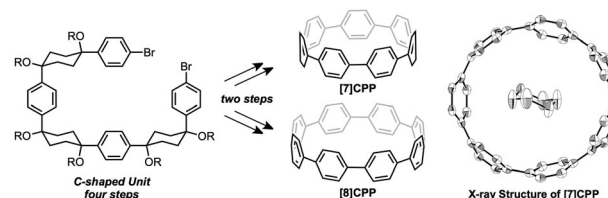


954

### Selective synthesis of [7]- and [8]cycloparaphenylenes

Friederike Sibbel, Katsuma Matsui, Yasutomo Segawa, Armido Studer and Kenichiro Itami\*

We report a size-selective synthesis of [7]- and [8]CPP by a modular approach and present the first X-ray crystal structure of [7]CPP.

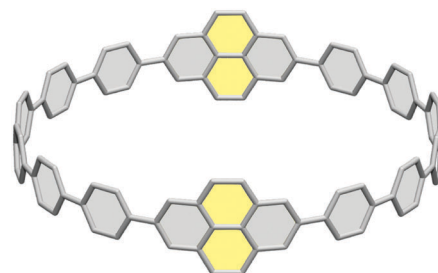


957

### Synthesis and properties of cycloparaphenylene-2,7-pyrenylene: a pyrene-containing carbon nanoring

Akiko Yagi, Gandikota Venkataramana, Yasutomo Segawa and Kenichiro Itami\*

The first synthesis of a pyrene-containing carbon nanoring, cyclo[12]paraphenylene[2]-2,7-pyrenylene ([12,2]CPPyr), is described.

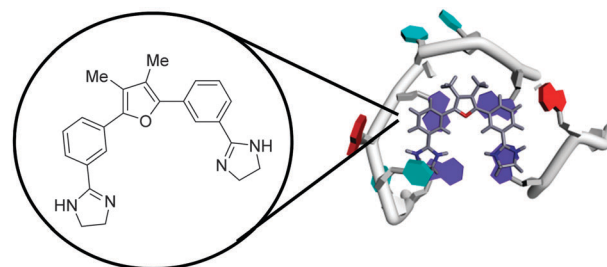
Pyrene-containing  $\pi$ -extended carbon nanoring

960

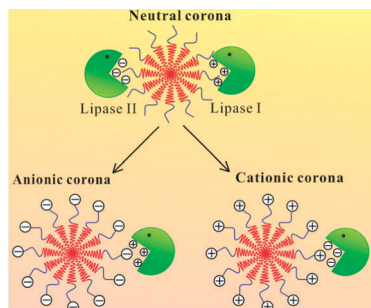
### Discovery of new G-quadruplex binding chemotypes

Stephan A. Ohnmacht, Ehsan Varavipour, Rupesh Nanjunda, Ingrida Pazitna, Gloria Di Vita, Mekala Gunaratnam, Arvind Kumar, Mohamed A. Ismail, David W. Boykin, W. David Wilson and Stephen Neidle\*

We report a novel furan-based low molecular weight chemotype with high G-quadruplex affinity and potent anti-proliferative activity.



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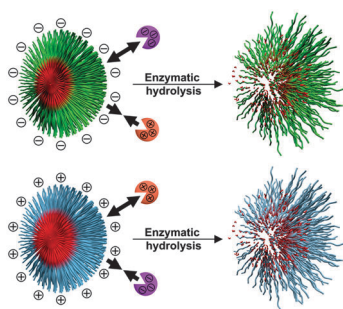


### Corona charge selective micelle degradation catalyzed by *P. cepacia* lipase isoforms

Xiaobo Zhu, Michael Fryd, Ann M. Valentine\* and Bradford B. Wayland\*

Kinetic studies of *P. cepacia* lipase catalyzed degradation of HO-PCL<sub>n</sub>-*b*-PEG<sub>32</sub>-RX block copolymer micelles with charged groups placed on the corona terminus resulted in detecting the presence of two lipase isoforms that manifest complementary micelle corona charge selective catalysis.

968

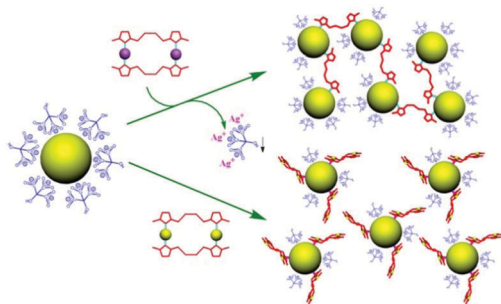


### Programmed hydrolysis of nanoassemblies by electrostatic interaction-mediated enzymatic-degradation

Sandani Samarajeewa, Ryan P. Zentay, Nema D. Jhurry, Ang Li, Kellie Seetho, Jiong Zou and Karen L. Wooley\*

At physiological pH, polymer nanoparticles having degradable-core and charged-shell morphologies readily attract or repel enzymes carrying opposite or similar charges, respectively.

971

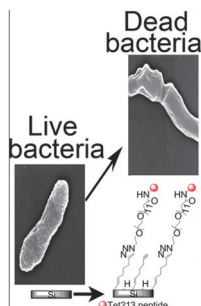


### Metallamacrocycle-modified gold nanoparticles: a new pathway for surface functionalization

Hai-Xia Liu, Xin He and Liang Zhao\*

We report herein the designed synthesis of four silver(i)- or gold(i)-bridged flexible metalla-macrocycles (MMCs) and their distinct performance in the surface modification of gold nanoparticles (AuNPs). The resulting gold(i)-MMCs-modified AuNPs were found to be resistant to pH variation and capable of binding with metal ions.

975



### The promotion of antimicrobial activity on silicon substrates using a "click" immobilized short peptide

Lin Wang, Junjian Chen, Lin Shi, Zhifeng Shi, Li Ren\* and Yingjun Wang\*

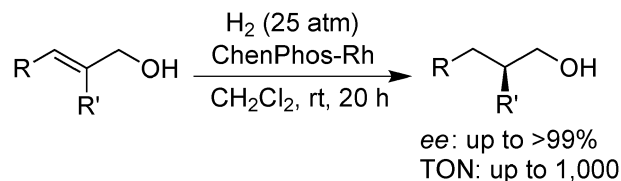
After being clicked with the Tet213 peptide, the Si surface could show excellent antimicrobial activity and good biocompatibility.

978

### Highly enantioselective hydrogenation of 2-substituted-2-alkenols catalysed by a ChenPhos–Rh complex

Quanjun Wang, Xueying Liu, Xian Liu, Bin Li, Huifang Nie, Shengyong Zhang\* and Weiping Chen\*

Highly enantioselective hydrogenation of a variety of 2-substituted-2-alkenols has been achieved using a ChenPhos–Rh complex as catalyst, giving  $\geq 99\%$  ee for most substrates.

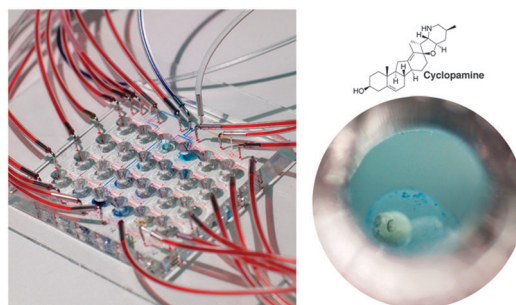


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### Fish in chips: an automated microfluidic device to study drug dynamics *in vivo* using zebrafish embryos

Chunhong Zheng, Hongwei Zhou, Xinxing Liu, Yuhong Pang, Bo Zhang and Yanyi Huang\*

A programmable and automated microfluidic device that allows dynamic monitoring of the *in vivo* assays.

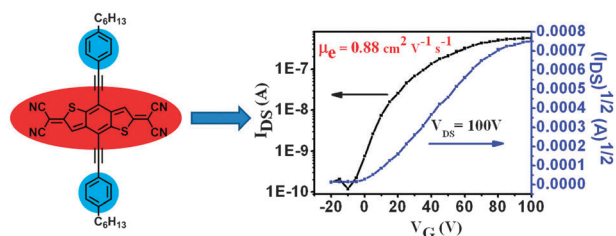


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### Donor–acceptor–donor type organic semiconductor containing quinoidal benzo[1,2-*b*:4,5-*b'*]dithiophene for high performance n-channel field-effect transistors

Shitao Wang, Mao Wang, Xu Zhang, Xiaodi Yang, Qiuli Huang, Xiaolan Qiao, Haixia Zhang, Qinghe Wu, Yu Xiong, Jianhua Gao and Hongxiang Li\*

A donor–acceptor–donor type organic semiconductor, with dicyano-substituted quinoidal benzo[1,2-*b*:4,5-*b'*]dithiophene as the acceptor, was synthesized and studied.

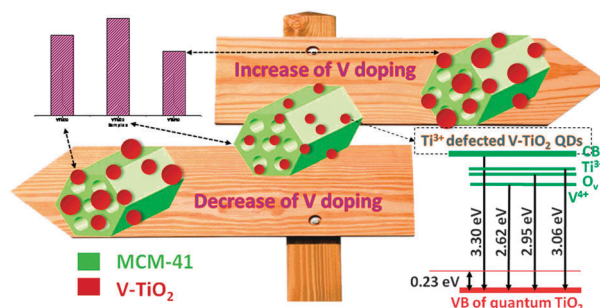


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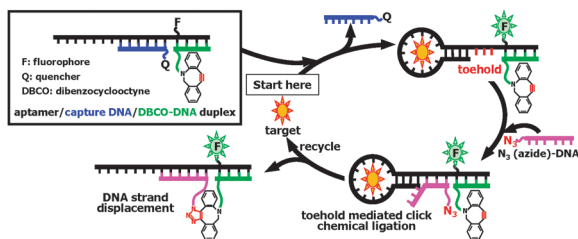
### Ti<sup>3+</sup>-defected and V-doped TiO<sub>2</sub> quantum dots loaded on MCM-41

Lun Pan, Songbo Wang, Ji-Jun Zou,\* Zhen-Feng Huang, Li Wang and Xiangwen Zhang

V-doped TiO<sub>2</sub> QDs with many Ti<sup>3+</sup> defects are fabricated by simply combining trace V doping and the loading strategy on MCM-41. Such QDs show significantly high photoactivity due to the quantum-size effect and the newly formed Ti<sup>3+</sup>- and V<sup>4+</sup>-mediated band levels.



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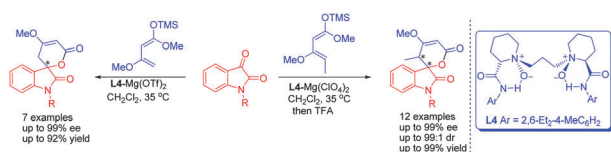


### Enzyme-free fluorescent-amplified aptasensors based on target-responsive DNA strand displacement *via* toehold-mediated click chemical ligation

Motoi Oishi,\* Shingo Nakao and Daiki Kato

A new target-responsive DNA strand displacement system *via* toehold-mediated click chemical ligation was designed and prepared for enzyme-free fluorescent-amplified aptasensors.

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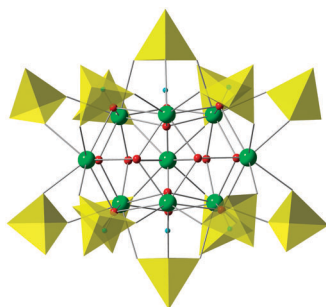


### Magnesium(II)-catalyzed asymmetric hetero-Diels reaction of Brassard's dienes with isatins

Jianfeng Zheng, Lili Lin, Yulong Kuang, Jiannan Zhao, Xiaohua Liu and Xiaoming Feng\*

The first catalytic asymmetric hetero-Diels–Alder reaction of Brassard's dienes with isatins was realized using Mg(II)/*N,N'*-dioxide complexes as catalysts, affording the corresponding chiral spirocyclic products bearing tetrasubstituted centers in up to 99% yield with up to 99% ee and >99:1 dr within 3 hours.

997

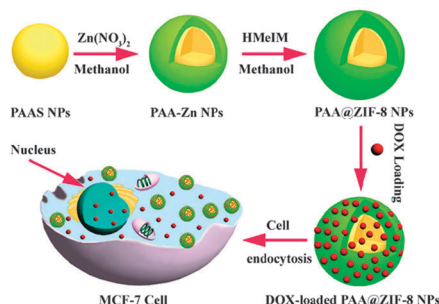


### A novel nonanuclear hafnium oxide–hydroxide–sulphate cluster crystallised from aqueous solution

A. Kalaji and L. Soderholm\*

Single crystals of  $(\text{NH}_4)_{14}[\text{Hf}_9\text{O}_8(\text{OH})_6(\text{SO}_4)_{14}] \cdot n\text{H}_2\text{O}$  (**1**) were obtained by heating a sealed aqueous solution of  $\text{HfOCl}_2 \cdot 8\text{H}_2\text{O}$ ,  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{H}_2\text{SO}_4$  at 80 °C for 10 days. The discrete  $[\text{Hf}_9\text{O}_8(\text{OH})_6(\text{SO}_4)_{14}]^{14-}$  anionic clusters have no inter-cluster connectivity. This rare nonameric architecture has only been observed previously in two  $\text{Bi}^{3+}$  oxo clusters.

1000



### Polyacrylic acid@zeolitic imidazolate framework-8 nanoparticles with ultrahigh drug loading capability for pH-sensitive drug release

Hong Ren, Lingyu Zhang, Jiping An, Tingting Wang,\* Lu Li, Xiaoyan Si, Liu He, Xiaotong Wu, Chungang Wang\* and Zhongmin Su\*

A facile route was developed to fabricate the polyacrylic acid@ZIF-8 nanoparticles (NPs), possessing ultrahigh doxorubicin loading capability ( $1.9 \text{ g DOX g}^{-1} \text{ NPs}$ ), which were employed as pH-dependent drug delivery vehicles.



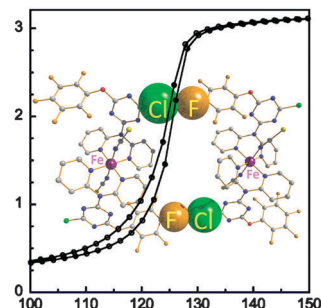
## COMMUNICATIONS

1003

**Enhancement of spin-crossover cooperativity mediated by lone pair– $\pi$  interactions and halogen bonding**

Nassim Nassirinia, Saeid Amani, Simon J. Teat, Olivier Roubeau\* and Patrick Gamez\*

Rational ligand design has allowed the generation of a highly cooperative spin-transition iron(II) complex, an unprecedented result in the family of (2,2'-dipyridylamino/s-triazine)-based SCO materials.

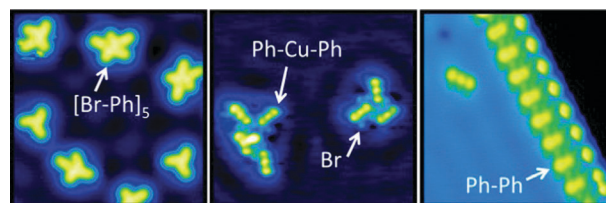


1006

**Atomic-scale insight into the formation, mobility and reaction of Ullmann coupling intermediates**

Emily A. Lewis, Colin J. Murphy, Melissa L. Liriano and E. Charles H. Sykes\*

Molecular-scale imaging reveals that the surface-catalyzed Ullmann coupling of bromobenzene occurs via a mobile organometallic intermediate composed of a single Cu atom complexed by two phenyl groups.

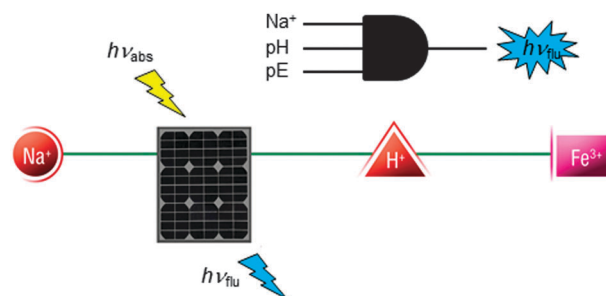


1009

**A sodium-enabled 'Pourbaix sensor': a three-input AND logic gate as a 'lab-on-a-molecule' for monitoring  $\text{Na}^+$ , pH and pE**

David C. Magri,\* Matthew Camilleri Fava and Carl J. Mallia

A three-input AND logic gate with a fluorescence output is demonstrated to be a 'lab-on-a-molecule' for monitoring  $\text{Na}^+$ , pH and pE in solution.

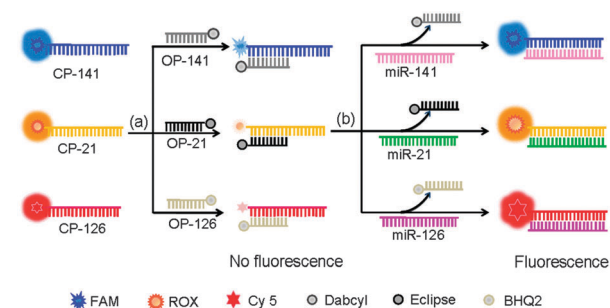


1012

**DNA strand-displacement-induced fluorescence enhancement for highly sensitive and selective assay of multiple microRNA in cancer cells**

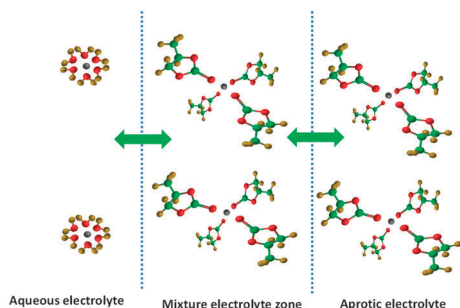
Ping Wu, Yunqiu Tu, Yingdan Qian, Hui Zhang and Chenxin Cai\*

A sensitive, selective, and nontarget-amplification method based on DNA strand-displacement-induced fluorescence enhancement for evaluating multiple miRNA expressions in cancer cells is reported.



## COMMUNICATIONS

1015

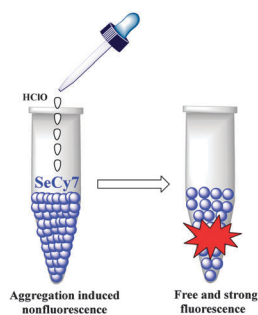


### Facilitated $\text{Li}^+$ ion transfer across the water/1,2-dichloroethane interface by the solvation effect

Uwitonze Nestor, Hanmei Wen, Girum Girma, Ziqiang Mei, Wenkai Fei, Yong Yang, Cunzhong Zhang\* and Dongping Zhan\*

We demonstrate that the solvation effect can be the driving force for ion transfer across the water/1,2-dichloroethane interface.

1018

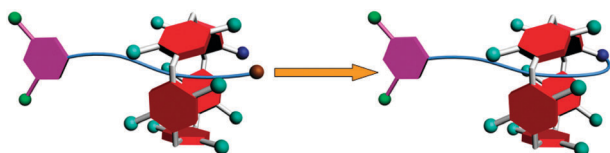


### A near-infrared fluorescent probe for selective detection of $\text{HClO}$ based on Se-sensitized aggregation of heptamethine cyanine dye

Guanghui Cheng, Jiangli Fan,\* Wen Sun, Jianfang Cao, Chong Hu and Xiaojun Peng\*

A Se-containing heptamethine cyanine dye based fluorescent probe was successfully developed and used for  $\text{HClO}$  detection with rapid response and high selectivity based on aggregation behavior. It is practical in detecting  $\text{HClO}$  in fetal bovine serum and living mice.

1021

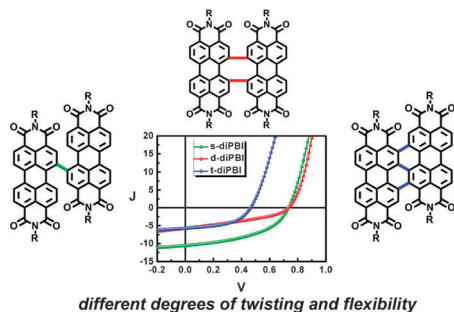


### Design and efficient synthesis of a pillar[5]arene-based [1]rotaxane

Binyuan Xia and Min Xue\*

The first pillararene-based [1]rotaxane was efficiently synthesized and investigated.

1024



### Bay-linked perylene bisimides as promising non-fullerene acceptors for organic solar cells

Wei Jiang, Long Ye, Xiangguang Li, Chengyi Xiao, Fang Tan, Wenchao Zhao, Jianhui Hou\* and Zhaohui Wang\*

A series of bay-linked perylene bisimides with the different degrees of twisting and flexibility are promising as non-fullerene acceptors for organic solar cells.

1027

### Highly specific enrichment of *N*-linked glycopeptides based on hydrazide functionalized soluble nanopolymers

Lijuan Zhang, Hucong Jiang, Jun Yao, Yali Wang, Caiyun Fang, Pengyuan Yang and Haojie Lu\*

Hydrazide functionalized PAMAM was synthesized for selective enrichment of *N*-linked glycopeptides from biological samples using FASP (filter-aided sample preparation) mode.

