

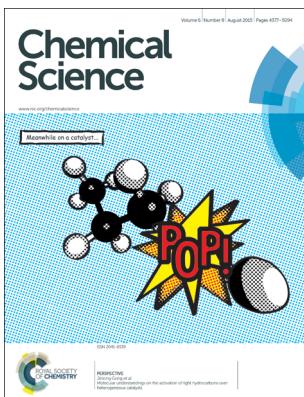
# Chemical Science

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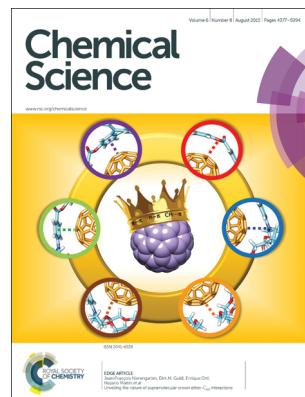
## IN THIS ISSUE

ISSN 2041-6539 CODEN CSHCBM 6(8) 4377–5094 (2015)



### Cover

See Jinlong Gong *et al.*, pp. 4403–4425.  
Image reproduced by permission of Jinlong Gong from *Chem. Sci.*, 2015, 6, 4403.



### Inside cover

See Jean-François Nierengarten, Dirk M. Guldin, Enrique Ortí, Nazario Martín *et al.*, pp. 4426–4432.  
Image reproduced by permission of Nazario Martín from *Chem. Sci.*, 2015, 6, 4426.

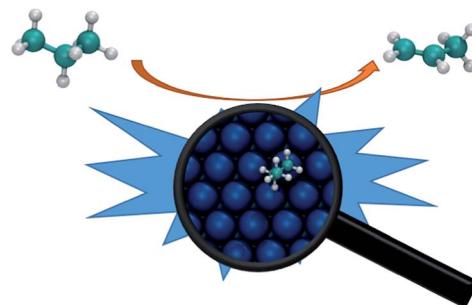
## PERSPECTIVE

4403

### Molecular understandings on the activation of light hydrocarbons over heterogeneous catalysts

Zhi-Jian Zhao, Cheng-chau Chiu and Jinlong Gong\*

This review describes recent progress on mechanistic understanding of heterogeneous catalytic dehydrogenation reactions of light alkanes.



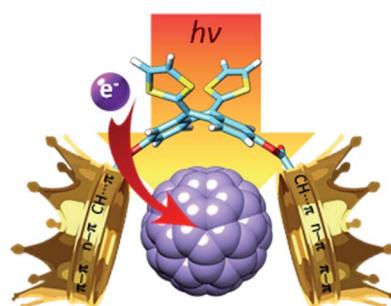
## EDGE ARTICLES

4426

### Unveiling the nature of supramolecular crown ether–C<sub>60</sub> interactions

L. Moreira, J. Calbo, R. M. Krick Calderon, J. Santos, B. M. Illescas, J. Aragó, J.-F. Nierengarten,\* D. M. Guldin,\* E. Ortí\* and N. Martín\*

Preparation of exTTF-(crown ether)<sub>2</sub> receptors, which host C<sub>60</sub>, to understand the nature of the fullerene–crown ether interaction. A combination of experimental and *in silico* studies suggest that it results from the interplay of donor–acceptor,  $\pi$ – $\pi$ , n– $\pi$  and CH··· $\pi$  interactions.



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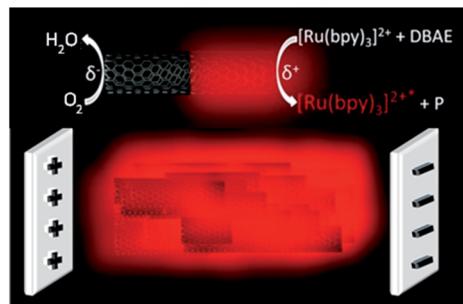
## EDGE ARTICLES

4433

**3D electrogenerated chemiluminescence: from surface-confined reactions to bulk emission**

Milica Sentic, Stéphane Arbault, Laurent Bouffier, Dragan Manojlovic, Alexander Kuhn\* and Neso Sojic\*

Electrogenerated chemiluminescence is extended to the 3D by generating light at the level of millions of micro-emitters addressed remotely by bipolar electrochemistry.

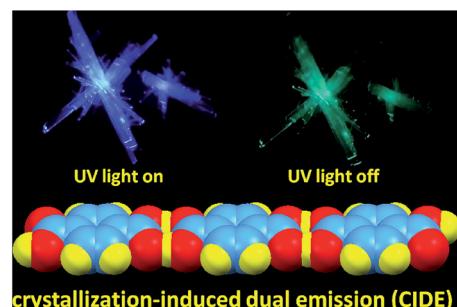


4438

**Crystallization-induced dual emission from metal- and heavy atom-free aromatic acids and esters**

Yongyang Gong, Lifang Zhao, Qian Peng, Di Fan, Wang Zhang Yuan,\* Yongming Zhang\* and Ben Zhong Tang

Crystallization-induced dual emission (fluorescence and phosphorescence) is observed in a group of pure organic aromatic acids and esters.

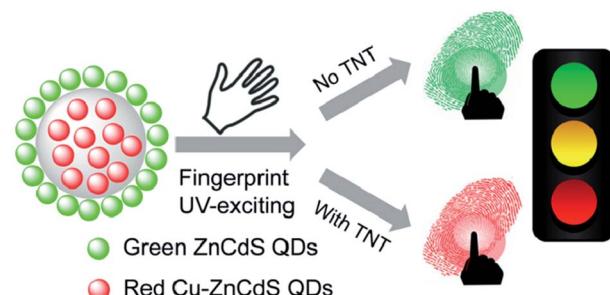


4445

**Dual-emitting quantum dot nanohybrid for imaging of latent fingerprints: simultaneous identification of individuals and traffic light-type visualization of TNT**

Peng Wu, Chaoying Xu, Xiandeng Hou, Jing-Juan Xu\* and Hong-Yuan Chen\*

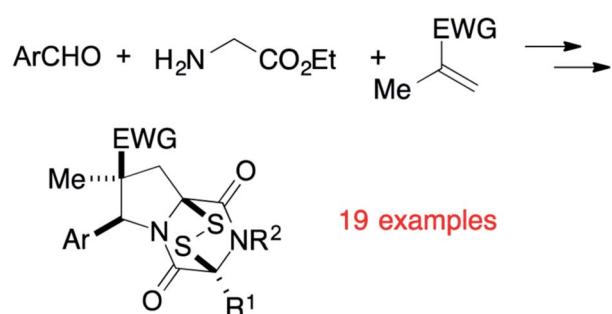
A nanohybrid was employed for fingerprint imaging that was capable of simultaneous identification of individuals and TNT visualization in a "traffic-light" manner.



4451

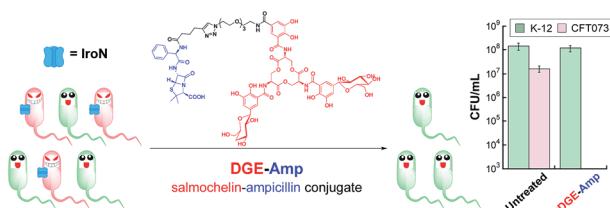
**Tricyclic analogues of epidithiodioxopiperazine alkaloids with promising *in vitro* and *in vivo* antitumor activity**

Marcus Baumann, André P. Dieskau, Brad M. Loertscher, Mary C. Walton, Sangkil Nam, Jun Xie, David Horne\* and Larry E. Overman\*

A short synthesis of 1,4-dioxohexahydro-6*H*-3,8*a*-epidithiopyrrolo[1,2-*a*]pyrazines will enable future mechanistic and translational studies of these structurally novel and promising clinical antitumor candidates.

## EDGE ARTICLES

4458



**Targeting virulence: salmochelin modification tunes the antibacterial activity spectrum of  $\beta$ -lactams for pathogen-selective killing of *Escherichia coli***

Phoom Chairatana, Tengfei Zheng and Elizabeth M. Nolan\*

New antibiotics are required to treat bacterial infections and counteract the emergence of antibiotic resistance.

4472

## Object Image

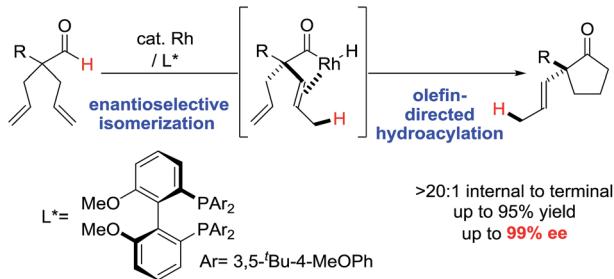


**Small molecular logic systems can draw the outlines of objects via edge visualization**

Jue Ling, Gaowa Naren, Jessica Kelly, David B. Fox and A. Prasanna de Silva

Like a child with a crayon, logical molecules produce outline drawings from a template.

4479

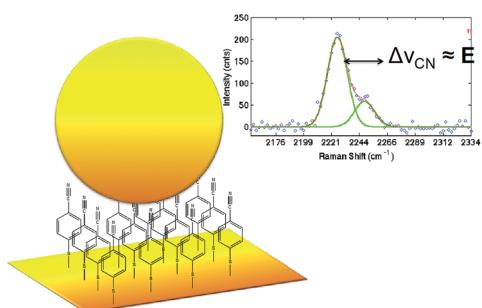


**Rh-catalyzed desymmetrization of  $\alpha$ -quaternary centers by isomerization-hydroacylation**

Jung-Woo Park, Kevin G. M. Kou, Daniel K. Kim and Vy M. Dong\*

A Rh-catalyzed desymmetrization of  $\alpha,\alpha$ -bis(allyl)aldehydes occurs by enantioselective isomerization followed by olefin-directed hydroacylation.

4484



**Alkyl-nitrile adlayers as probes of plasmonically induced electric fields**

Daniel T. Kwasnieski, Hao Wang and Zachary D. Schultz\*

Adsorbed mercaptoalkynitriles show Stark shifts on plasmonic surfaces that provide a direct measurement of the induced electric field.



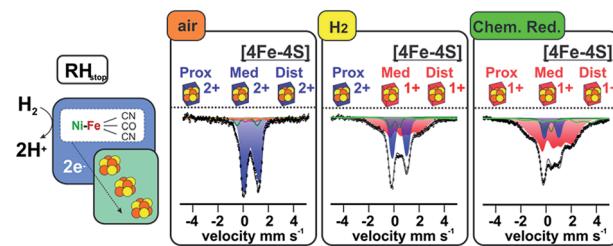
## EDGE ARTICLES

4495

**Cofactor composition and function of a H<sub>2</sub>-sensing regulatory hydrogenase as revealed by Mössbauer and EPR spectroscopy**

Federico Roncaroli, Eckhard Bill,\* Bärbel Friedrich, Oliver Lenz, Wolfgang Lubitz\* and Maria-Eirini Pandelia\*

A regulatory hydrogenase is characterised by Mössbauer, EPR and FTIR yielding insight into structure and function of this dihydrogen sensor.

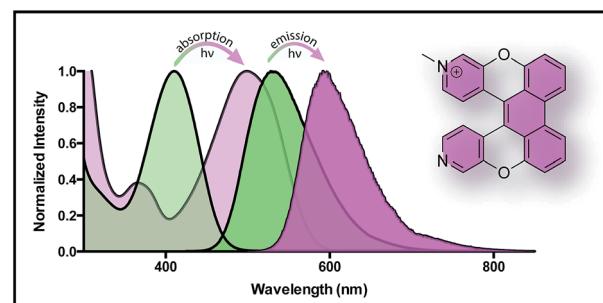


4508

**Synthesis and properties of lysosome-specific photoactivatable probes for live-cell imaging**

Mai N. Tran, Robert-André F. Rarig and David M. Chenoweth\*

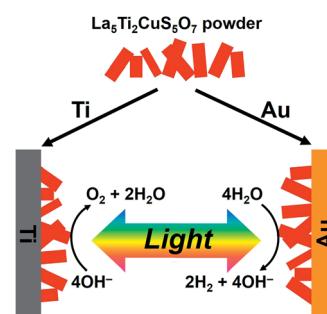
We describe the synthesis and application of a new class of large Stokes shift lysosome-specific photoactivatable probes for live-cell imaging.



4513

**Photoanodic and photocathodic behaviour of La<sub>5</sub>Ti<sub>2</sub>CuS<sub>5</sub>O<sub>7</sub> electrodes in the water splitting reaction**

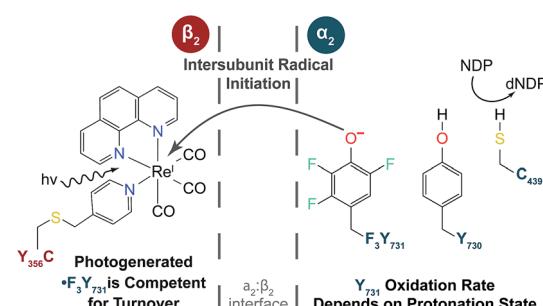
Guojun Ma, Yohichi Suzuki, Rupashree Balia Singh, Aki Iwanaga, Yosuke Moriya, Tsutomu Minegishi, Jingyuan Liu, Takashi Hisatomi, Hiroshi Nishiyama, Masao Katayama, Kazuhiko Seki, Akihiro Furube, Taro Yamada and Kazunari Domen\*

La<sub>5</sub>Ti<sub>2</sub>CuS<sub>5</sub>O<sub>7</sub> embedded into the surface of Au and Ti substrates shows a photocurrent attributable to HER and OER.

4519

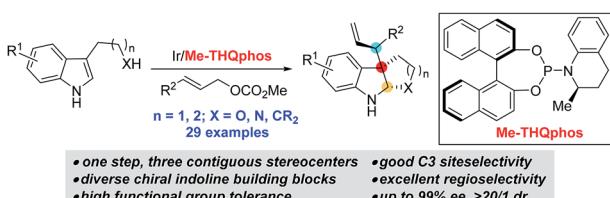
**Direct interfacial Y<sub>731</sub> oxidation in  $\alpha_2$  by a photo $\beta_2$  subunit of *E. coli* class Ia ribonucleotide reductase**

David Y. Song, Arturo A. Pizano, Patrick G. Holder, JoAnne Stubbe\* and Daniel G. Nocera\*

Proton-coupled electron transfer (PCET) is a fundamental mechanism important in a wide range of biological processes including the universal reaction catalysed by ribonucleotide reductases (RNRs) in making *de novo*, the building blocks required for DNA replication and repair.

## EDGE ARTICLES

4525

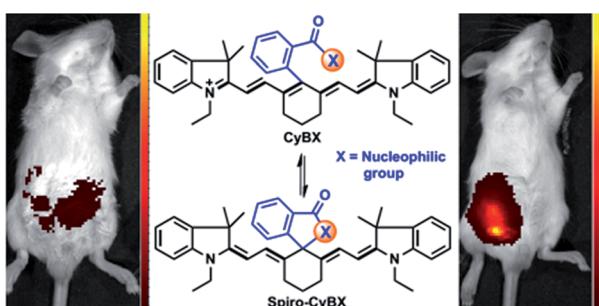


### Ligand-enabled Ir-catalyzed intermolecular diastereoselective and enantioselective allylic alkylation of 3-substituted indoles

Xiao Zhang, Wen-Bo Liu, Hang-Fei Tu and Shu-Li You\*

A ligand-enabled Ir-catalyzed diastereoselective and enantioselective allylic alkylation of 3-substituted indoles is reported, providing indoline products containing three contiguous stereocenters in one step with high site-, regio-, diastereo- and enantioselectivities from a wide range of readily available starting materials.

4530

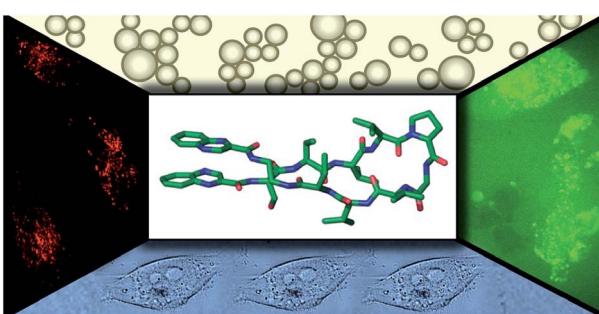


### A simple and effective "capping" approach to readily tune the fluorescence of near-infrared cyanines

Longwei He, Weiyi Lin,\* Qiuyan Xu, Mingguang Ren, Haipeng Wei and Jian-Yong Wang

A simple and effective capping approach was introduced to readily tune the fluorescence of NIR cyanines.

4537

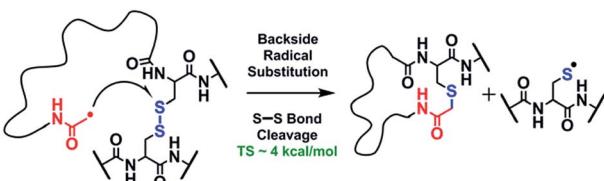


### A new quinoxaline-containing peptide induces apoptosis in cancer cells by autophagy modulation

Rubí Zamudio-Vázquez, Saška Ivanova, Miguel Moreno, María Isabel Hernández-Alvarez, Ernest Giralt, Axel Bidon-Chanal, Antonio Zorzano,\* Fernando Albericio\* and Judit Tulla-Puche\*

The most cytotoxic compound from a library of quinoxaline-containing peptides is endocytosed into HeLa cells, accumulates in acidic compartments, and blocks autophagy by altering lysosomal function, leading to apoptosis activation.

4550



### Mechanisms and energetics of free radical initiated disulfide bond cleavage in model peptides and insulin by mass spectrometry

Chang Ho Sohn, Jinshan Gao, Daniel A. Thomas, Tae-Young Kim, William A. Goddard III and J. L. Beauchamp\*

Direct radical substitution at sulfur initiates disulfide bond cleavage by hydrogen-deficient radicals in peptides and proteins.

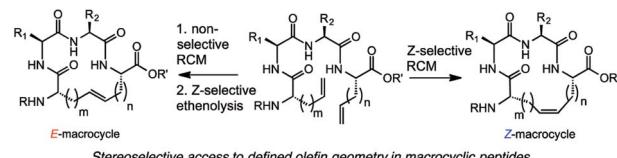


4561

# Stereoselective synthesis of macrocyclic peptides via a dual olefin metathesis and ethenolysis approach

Shane L. Mangold and Robert H. Grubbs\*

A metathesis strategy for controlling olefin geometry within macrocyclic peptides has been achieved using catalyst-directed RCM and ethenolysis.

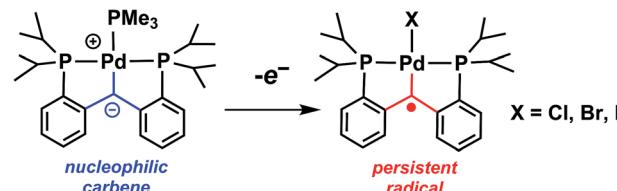


4570

## Palladium carbene complexes as persistent radicals

C. C. Comanescu, M. Vyushkova and V. M. Iluc\*

A series of palladium(II) persistent radical carbene complexes,  $[\text{PC}(\text{sp}^2)\text{P}] \text{PdX}$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ), was synthesized from the nucleophilic carbene  $[\text{PC}(\text{sp}^2)\text{P}] \text{PdPMes}_3$ .



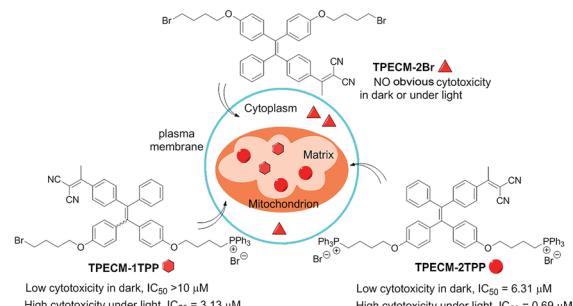
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4580

# Image-guided combination chemotherapy and photodynamic therapy using a mitochondria-targeted molecular probe with aggregation-induced emission characteristics

Chong-Jing Zhang, Qinglian Hu, Guangxue Feng,  
Ruoyu Zhang, Youyong Yuan, Xianmao Lu and Bin Liu\*

# Mitochondria-targeted AIE photosensitizers show multifunctions of targeted and image-guided combination chemotherapy and photodynamic therapy



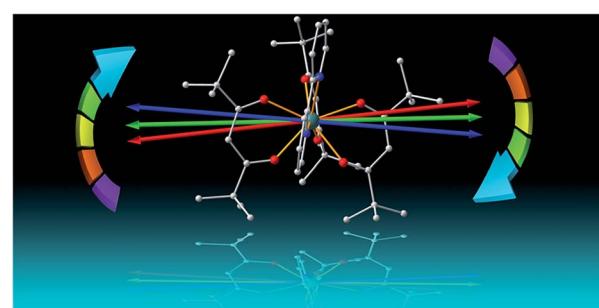
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4587

## Does the thermal evolution of molecular structures critically affect the magnetic anisotropy?

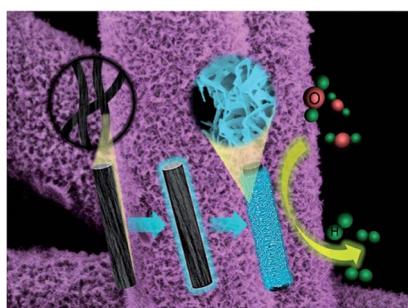
Kang Qian, José J. Baldoví, Shang-Da Jiang,\* Alejandro Gaita-Ariño,\* Yi-Quan Zhang, Jacob Overgaard, Bing-Wu Wang, Eugenio Coronado\* and Song Gao\*

In the absence of a critical phase transition, one can safely use the crystal structure information determined at liquid nitrogen temperature in magnetic anisotropy research.



## EDGE ARTICLES

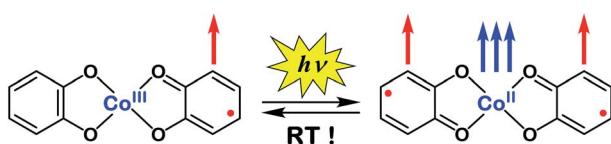
4594


**Cobalt diselenide nanobelts grafted on carbon fiber felt: an efficient and robust 3D cathode for hydrogen production**

Ya-Rong Zheng, Min-Rui Gao, Zi-You Yu, Qiang Gao, Huai-Ling Gao and Shu-Hong Yu\*

An easily scaled-up 3D CoSe<sub>2</sub>/CFF hierarchical electrode has been developed as a highly active and stable hydrogen evolution reaction cathode.

4599


**Bidirectional photoswitching of magnetic properties at room temperature: ligand-driven light-induced valence tautomerism**

Alexander Witt, Frank W. Heinemann and Marat M. Khusniyarov\*

A unique molecular switch – a cobalt dioxolene complex featuring photoisomerizable ligands – allows unprecedented control of magnetic properties with light at room temperature.

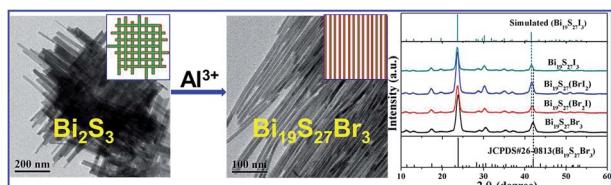
4610


**Oxalyl amide assisted palladium-catalyzed synthesis of pyrrolidones via carbonylation of  $\gamma$ -C(sp<sup>3</sup>)-H bonds of aliphatic amine substrates**

Chao Wang, Li Zhang, Changpeng Chen, Jian Han, Yingming Yao\* and Yingsheng Zhao\*

The first Pd-catalyzed regioselective  $\gamma$ -carbonylation of oxalyl amide protected aliphatic amines with carbon monoxide leading to synthesis of pyrrolidones has been developed.

4615


**Shape and composition control of Bi<sub>19</sub>S<sub>27</sub>(Br<sub>3-x</sub>I<sub>x</sub>) alloyed nanowires: the role of metal ions**

Yihui Wu, Huanhuan Pan, Xin Zhou, Mingrun Li, Bin Zhou, Chi Yang, Wen-Hua Zhang,\* Jiansheng Jie\* and Can Li\*

Highly uniform single-crystalline Bi<sub>19</sub>S<sub>27</sub>(Br<sub>3-x</sub>I<sub>x</sub>) alloyed nanowires (NWs) (0 ≤ x ≤ 3) were achieved for the first time. The NWs show a composition-independent band gap and have great application potential in optoelectronic devices.

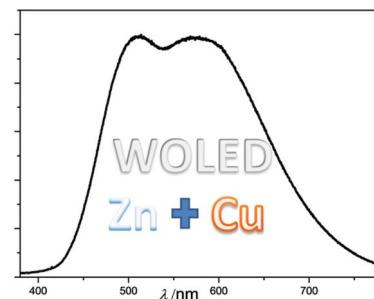
## EDGE ARTICLES

4623

**Luminescent zinc(II) and copper(I) complexes for high-performance solution-processed monochromic and white organic light-emitting devices**

G. Cheng, G. K.-M. So, W.-P. To, Y. Chen, C.-C. Kwok, C. Ma, X. Guan, X. Chang, W.-M. Kwok and C.-M. Che\*

High performance orange (EQE up to 15.64%) and white (EQE up to 6.88%) solution processed OLEDs fabricated solely with emitters of non-platinum group metals were reported. The white device has CIE coordinates of (0.42, 0.44) and CRI of 81.

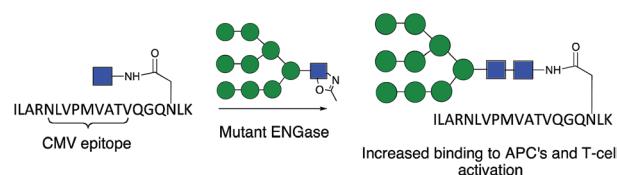


4636

**Convergent chemo-enzymatic synthesis of mannosylated glycopeptides; targeting of putative vaccine candidates to antigen presenting cells**

Julie D. McIntosh, Margaret A. Brimble,\* Anna E. S. Brooks, P. Rod Dunbar,\* Renata Kowalczyk, Yusuke Tomabechi and Antony J. Fairbanks\*

Convergent chemo-enzymatic synthesis of mannosylated glycopeptides enhances uptake by human antigen presenting cells whilst preserving the immunogenicity of peptide epitopes.

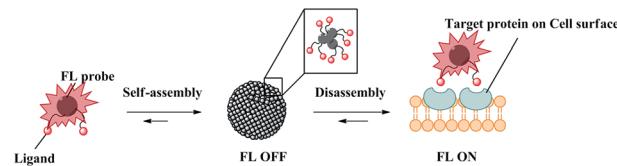


4643

**Near-infrared fluorescence activation probes based on disassembly-induced emission cyanine dye**

Tai-Cheng Hou, Ying-Yi Wu, Po-Yi Chiang and Kui-Thong Tan\*

In the presence of target analyte, bright fluorescence in the near-IR region is emitted through the recognition-induced disassembly of the probe aggregate.

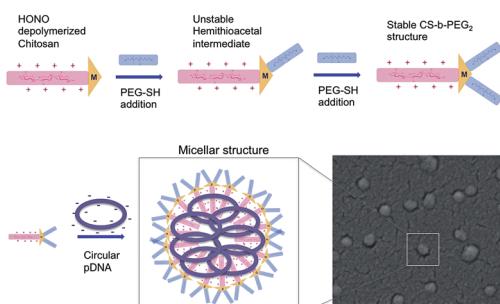


4650

**Regioselective thioacetylation of chitosan end-groups for nanoparticle gene delivery systems**

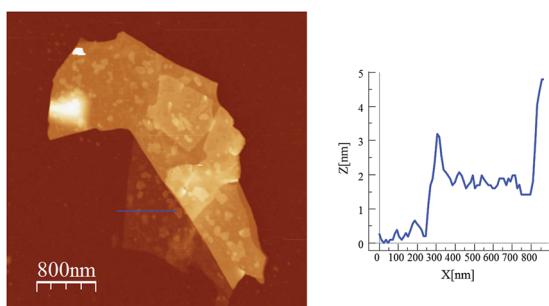
V. D. Pickenhahn, V. Darras, F. Dziopa, K. Biniecki, G. De Crescenzo, M. Lavertu\* and M. D. Buschmann\*

We present a novel, aqueous thiol-based conjugation strategy that constitutes an alternative to the oxime-click pathway for generating a reactive end-group on chitosan (CS), which could also be applicable to other polymers.



## EDGE ARTICLES

4665

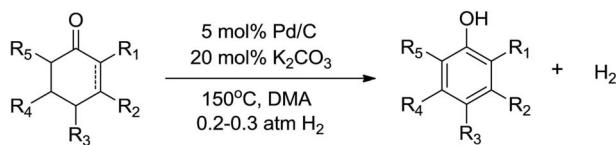


**Graphene related magnetic materials: micromechanical exfoliation of 2D layered magnets based on bimetallic anilate complexes with inserted  $[\text{Fe}^{\text{III}}(\text{acac}_2\text{-trien})]^+$  and  $[\text{Fe}^{\text{III}}(\text{sal}_2\text{-trien})]^+$  molecules**

Alexandre Abhervé, Samuel Mañas-Valero, Miguel Clemente-León\* and Eugenio Coronado\*

The Scotch tape method has been used for the exfoliation of layered coordination compounds formed by a 2D bimetallic anilate-based anionic network and  $\text{Fe}^{\text{III}}$  cationic complexes placed between or within the layers.

4674

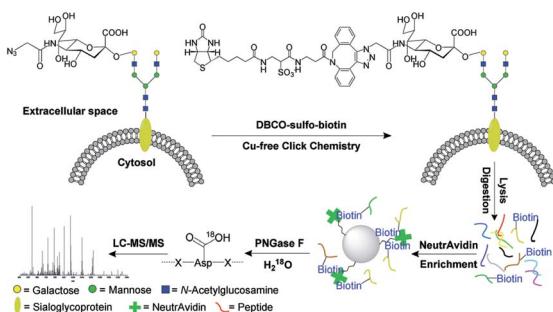


**Reaction-activated palladium catalyst for dehydrogenation of substituted cyclohexanones to phenols and  $\text{H}_2$  without oxidants and hydrogen acceptors**

Jingwu Zhang, Qiangqiang Jiang, Dejun Yang, Xiaomei Zhao, Yanli Dong and Renhua Liu\*

A combination of  $\text{Pd/C}$  and  $\text{H}_2$  is found to dehydrogenate a wide range of substituted cyclohexanones and 2-cyclohexenones to their corresponding phenols with high isolated yields, with  $\text{H}_2$  as the only byproduct.

4681

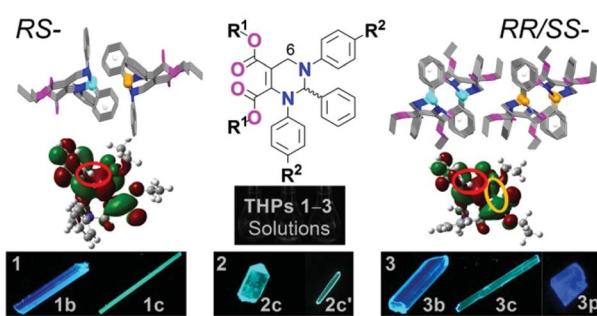


**Systematic and site-specific analysis of *N*-sialoglycosylated proteins on the cell surface by integrating click chemistry and MS-based proteomics**

Weixuan Chen, Johanna M. Smeekens and Ronghu Wu\*

A method integrating metabolic labeling, copper-free click chemistry and MS-based proteomics is effective to globally and site-specifically analyze surface *N*-sialoglycoproteins.

4690



**Insight into the strong aggregation-induced emission of low-conjugated racemic C6-unsubstituted tetrahydropyrimidines through crystal-structure–property relationship of polymorphs**

Qiuhua Zhu, Yilin Zhang, Han Nie, Zujin Zhao, Shuwen Liu,\* Kam Sing Wong\* and Ben Zhong Tang\*

Racemic low-conjugated non-emissive THPs 1–3 can form highly emissive *RS*- and *RR/SS*-packing polymorphs with mixed through-bond and through-space conjugation.



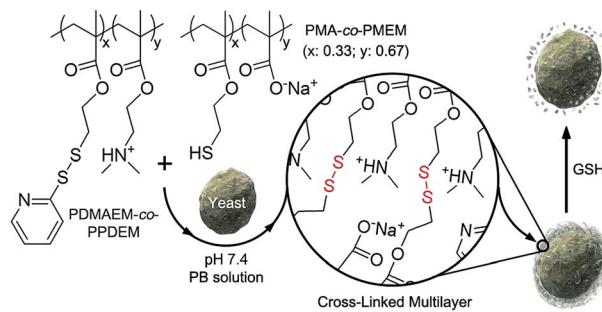
## EDGE ARTICLES

4698

**Cytocompatible *in situ* cross-linking of degradable LbL films based on thiol–exchange reaction**

Sung Ho Yang,\* Jinsu Choi, L. Palanikumar, Eun Seong Choi, Juno Lee, Juan Kim, Insung S. Choi\* and Ja-Hyoung Ryu\*

A highly cytocompatible LbL process was developed, based on the thiol–exchange reaction, for forming *in situ* cross-linked and degradable films under physiologically mild conditions.

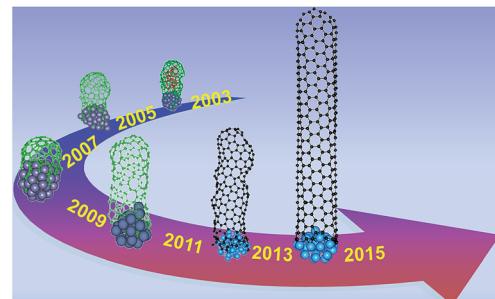


4704

**Atomistic simulation of the growth of defect-free carbon nanotubes**

Ziwei Xu, Tianying Yan\* and Feng Ding\*

The atomistic simulation of defect-free SWCNT growth is realized for the first time after 12 years of continuous effort.

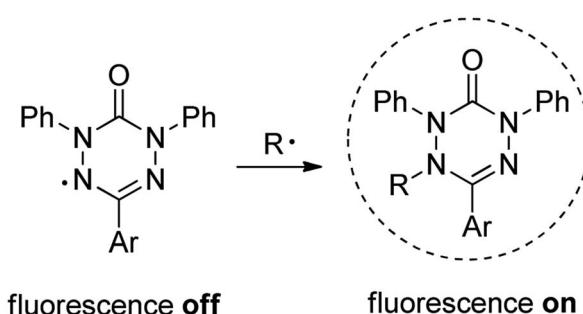


4712

**Profluorescent verdazyl radicals – synthesis and characterization**

David Matuschek, Steffen Eusterwiemann, Linda Stegemann, Carsten Doerenkamp, Birgit Wibbeling, Constantin G. Daniiliuc, Nikos L. Doltsinis,\* Cristian A. Strassert,\* Hellmut Eckert\* and Armido Studer\*

The synthesis and characterization of various 6-oxo-verdazyl radicals and their diamagnetic styryl radical trapping products are presented.

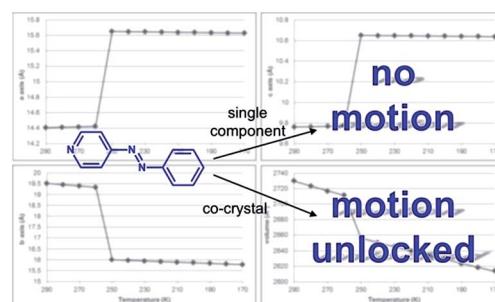


4717

**Achieving dynamic behaviour and thermal expansion in the organic solid state via co-crystallization**

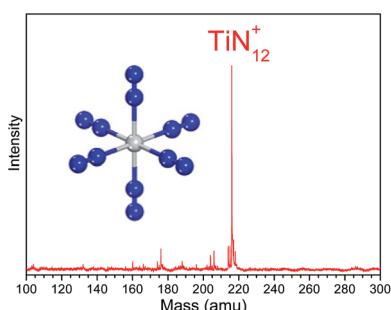
Kristin M. Hutchins, Ryan H. Groeneman, Eric W. Reinheimer, Dale C. Swenson and Leonard R. MacGillivray\*

Molecular motion of an azo functional group is 'unlocked' via co-crystallizations.



## EDGE ARTICLES

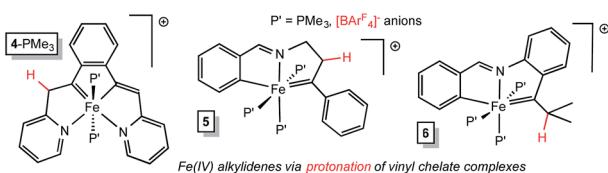
4723


**Experimental observation of  $\text{TiN}_{12}^+$  cluster and theoretical investigation of its stable and metastable isomers**

Ke-Wei Ding, Xiao-Wei Li, Hong-Guang Xu, Tao-Qi Li,\* Zhong-Xue Ge,\* Qian Wang\* and Wei-Jun Zheng\*

 In the  $O_h$  symmetric structure of  $\text{TiN}_{12}^+$ , the interaction between Ti and N<sub>2</sub> weakens the N–N bond significantly.

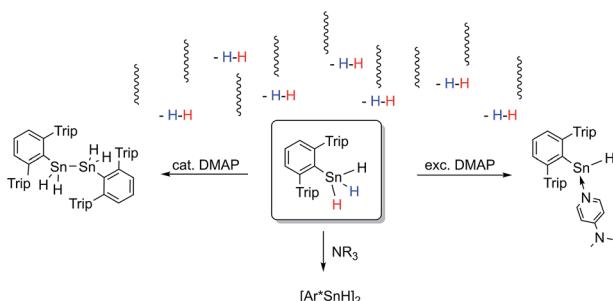
4730


**Fe(IV) alkylidenes *via* protonation of Fe(II) vinyl chelates and a comparative Mössbauer spectroscopic study**

Brian M. Lindley, Ala'aeddeen Swidan, Emil B. Lobkovsky, Peter T. Wolczanski,\* Mario Adelhardt, Jörg Sutter and Karsten Meyer

 Fe(IV) alkylidenes are produced *via* protonation of Fe(II) vinyl chelate complexes.

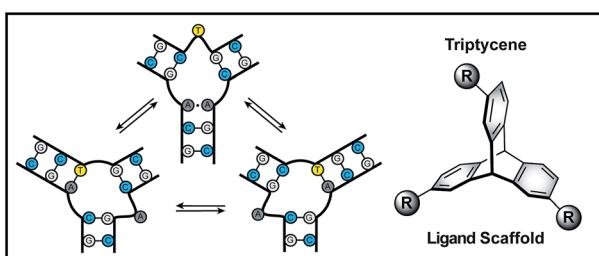
4737


**A nitrogen-base catalyzed generation of organotin(II) hydride from an organotin trihydride under reductive dihydrogen elimination**

Christian P. Sindlinger, Andreas Stasch, Holger F. Bettinger and Lars Wesemann\*

Amine bases are shown to induce reductive elimination of dihydrogen from terphenyltin trihydride.

4752


**Triptycene-based small molecules modulate (CAG)·(CTG) repeat junctions**

Stephanie A. Barros and David M. Chenoweth\*

A triptycene-based scaffold is used to develop a new class of ligands for modulating the structure of junction forming trinucleotide repeat expansion sequences.



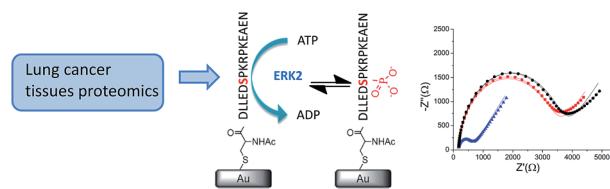
## EDGE ARTICLES

4756

**Integrating proteomics with electrochemistry for identifying kinase biomarkers**

Einav Amit, Rofeamor Obena, Yi-Ting Wang, Roman Zhuravel, Aaron James F. Reyes, Shir Elbaz, Dvir Rotem, Danny Porath, Assaf Friedler,\* Yu-Ju Chen\* and Shlomo Yitzchaik\*

We present an integrated approach for highly sensitive identification and validation of substrate-specific kinases as cancer biomarkers.

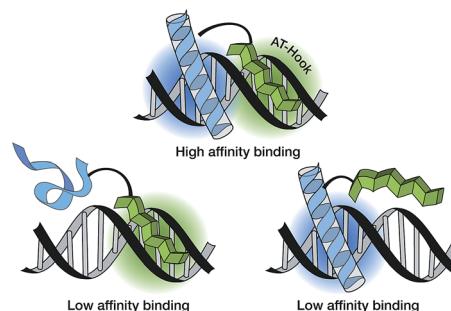


4767

**The AT-Hook motif as a versatile minor groove anchor for promoting DNA binding of transcription factor fragments**

Jéssica Rodríguez, Jesús Mosquera, Jose R. Couceiro, M. Eugenio Vázquez\* and José L. Mascareñas\*

We report the development of chimeric DNA binding peptides comprising a DNA binding fragment of natural transcription factors (the basic region of a bZIP protein or a monomeric zinc finger module) and an AT-Hook peptide motif.

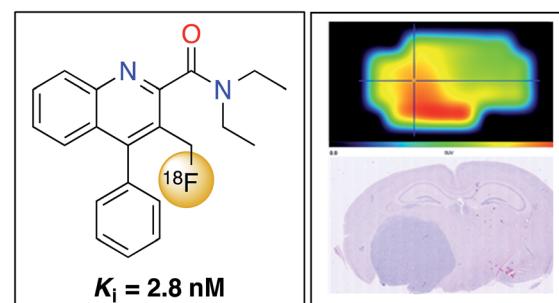


4772

**A novel <sup>18</sup>F-labelled high affinity agent for PET imaging of the translocator protein**

Adele Blair, Filip Zmuda, Gaurav Malviya, Adriana A. S. Tavares, Gilles D. Tamagnan, Anthony J. Chalmers, Deborah Dewar, Sally L. Pimlott and Andrew Sutherland\*

A novel <sup>18</sup>F-labelled quinoline-2-carboxamide has been characterised as a novel PET imaging agent for the translocator protein.

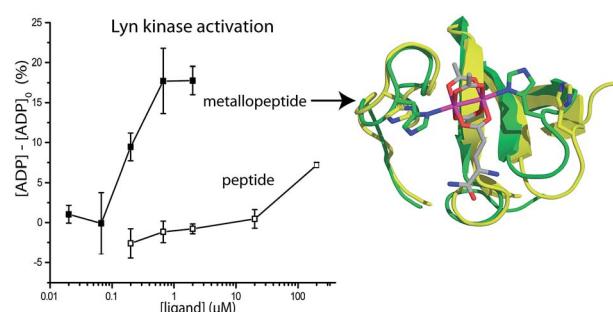


4778

**Potent and selective inhibition of SH3 domains with dirhodium metalloinhibitors**

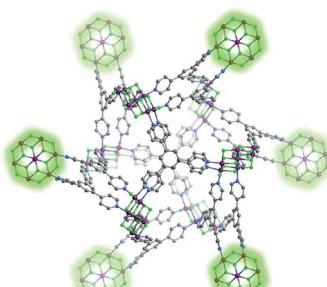
Farrukh Vohidov, Sarah E. Knudsen, Paul G. Leonard, Jun Ohata, Michael J. Whealon, Brian V. Popp, John E. Ladbury and Zachary T. Ball\*

Specific, designed histidine–rhodium interactions allow a metallopeptide to bind Lyn kinase with nanomolar affinity and to activate kinase activity.



## EDGE ARTICLES

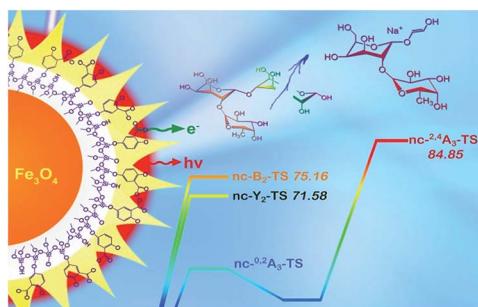
4784


**Double-walled pyr topology networks from a novel fluoride-bridged heptanuclear metal cluster**

Kai-Jie Chen, John J. Perry IV, Hayley S. Scott, Qing-Yuan Yang and Michael J. Zavorotko\*

Two isostructural networks with double-walled pyr topology comprised of novel fluoride-bridged heptanuclear metal clusters and 3-connected ligands have been synthesized and characterized by X-ray diffraction, thermogravimetric analysis, and gas sorption experiments.

4790


**UV-activated multilayer nanomatrix provides one-step tunable carbohydrate structural characterization in MALDI-MS**

Rofeamor P. Obena, Mei-Chun Tseng, Indah Primadona, Jun Hsiao, I-Che Li, Rey Y. Capangpangan, Hsiu-Fong Lu, Wan-Sheung Li, Ito Chao, Chun-Cheng Lin and Yu-Ju Chen\*

Our work highlights DHB@MNP-induced pseudo-MS/MS for oligosaccharide characterization, with some insights on this nanoparticle-mediated energy transfer dynamics.

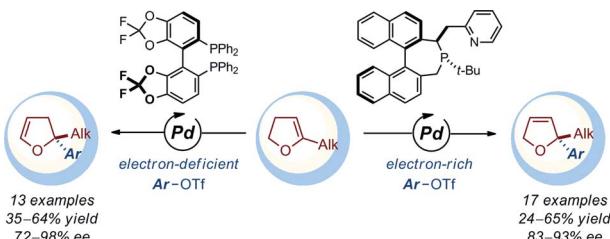
4801


**High fidelity sorting of remarkably similar components via metal-mediated assembly**

Lauren R. Holloway, Michael C. Young, Gregory J. O. Beran and Richard J. Hooley\*

Subtle differences in coordination angle and rigidity lead to narcissistic self-sorting between highly similar individual components upon metal-mediated assembly.

4807


**Access to enantioenriched 2,3- and 2,5-dihydrofurans with a fully substituted C2 stereocenter by Pd-catalyzed asymmetric intermolecular Heck reaction**

Gustavo M. Borrajo-Calleja, Vincent Bizet, Thomas Bürgi and Clément Mazet\*

A palladium catalyzed intermolecular asymmetric Heck reaction provides access to valuable 2,3- and 2,5-dihydrofurans with a fully substituted C2 stereocenter with high levels of regio- and enantiocontrol.



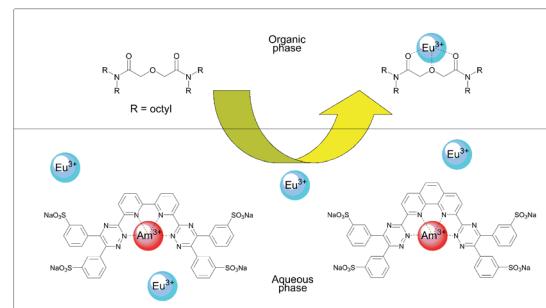
## EDGE ARTICLES

4812

**Hydrophilic sulfonated bis-1,2,4-triazine ligands are highly effective reagents for separating actinides(III) from lanthanides(III) via selective formation of aqueous actinide complexes**

Frank W. Lewis,\* Laurence M. Harwood,\* Michael J. Hudson, Andreas Geist, Valery N. Kozhevnikov, Petr Distler and Jan John

Tetrasulfonated bis-1,2,4-triazine ligands can selectively complex and separate actinides from lanthanides in aqueous nitric acid with very high selectivities.

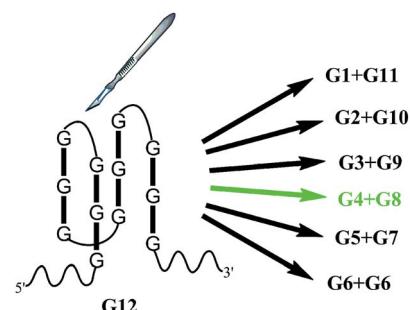


4822

**How to split a G-quadruplex for DNA detection: new insight into the formation of DNA split G-quadruplex**

Jinbo Zhu, Libing Zhang, Shaojun Dong and Erkang Wang\*

A magic "law of 4 : 8" to split the G-quadruplex for DNA detection has been found.

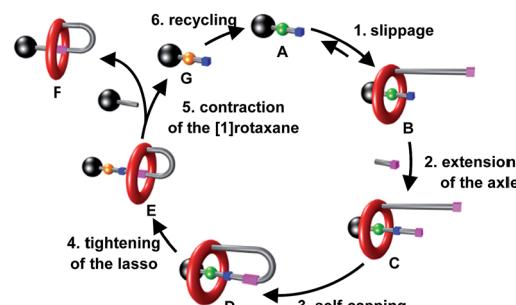


4828

**Synthesis of triazolium-based mono- and tri-branched [1]rotaxanes using a molecular transporter of dibenzo-24-crown-8**

P. Waelès, C. Clavel, K. Fournel-Marotte and F. Coutrot\*

A general synthesis to mono- and multi-branched [1]rotaxanes that are devoid of any efficient template is reported.

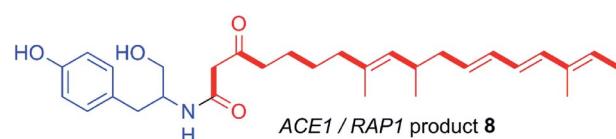


4837

**Heterologous expression of the avirulence gene *ACE1* from the fungal rice pathogen *Magnaporthe oryzae***

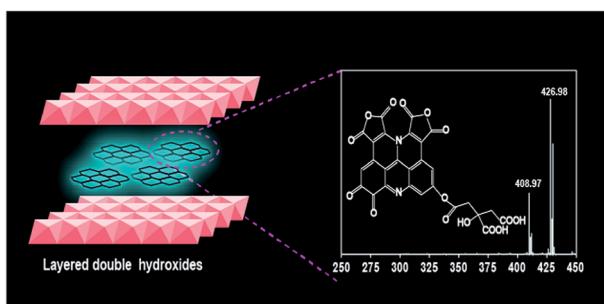
Z. Song, W. Bakeer, J. W. Marshall, A. A. Yakasai, R. M. Khalid, J. Collemare, E. Skellam, D. Tharreau, M.-H. Lebrun, C. M. Lazarus, A. M. Bailey, T. J. Simpson and R. J. Cox\*

Heterologous expression of key components of the *Magnaporthe grisea* *ACE1* gene cluster produces a potential precursor of cryptic avirulence signalling compounds that induce resistance to *M. grisea* in rice.



## EDGE ARTICLES

4846

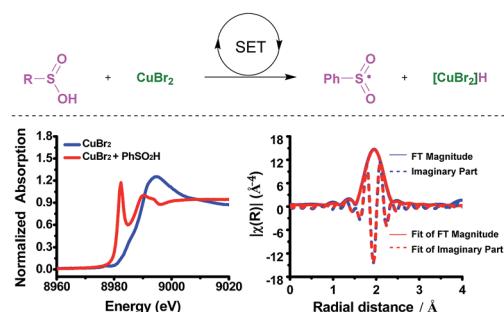


### Structure observation of graphene quantum dots by single-layered formation in layered confinement space

Liqing Song, Jingjing Shi, Jun Lu and Chao Lu\*

We observe the structure of single-layered graphene quantum dots prepared in the 2D confined space of layered double hydroxides.

4851

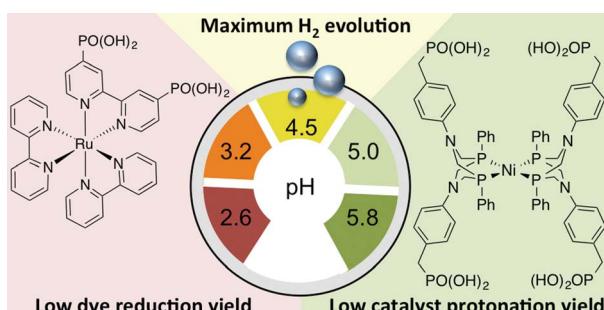


### Operando X-ray absorption and EPR evidence for a single electron redox process in copper catalysis

Qingquan Lu, Jian Zhang, Pan Peng, Guanghui Zhang, Zhiliang Huang, Hong Yi, Jeffrey T. Miller and Aiwen Lei\*

A single electron redox process between Cu(II) and a sulfenic acid, and characterization of the formed Cu(I) are clearly shown using *operando* X-ray absorption and EPR evidence.

4855

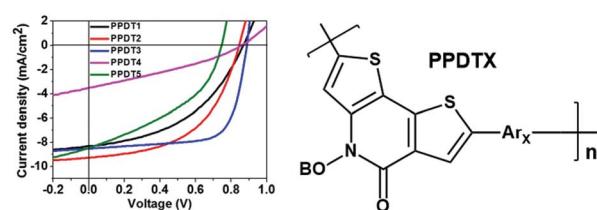


### Unravelling the pH-dependence of a molecular photocatalytic system for hydrogen production

Anna Reynal,\* Ernest Pastor, Manuela A. Gross, Shababa Selim, Erwin Reisner\* and James R. Durrant\*

The electron-donating ability of the sacrificial agent and the protonation of the catalyst determine the optimum pH for hydrogen production.

4860



### Wide bandgap OPV polymers based on pyridinonedithiophene unit with efficiency >5%

Alexander M. Schneider, Luyao Lu, Eric F. Manley, Tianyue Zheng, Valerii Sharapov, Tao Xu, Tobin J. Marks, Lin X. Chen\* and Luping Yu\*

We report the properties of a new series of wide band gap photovoltaic polymers based on the *N*-alkyl 2-pyridone dithiophene (PDT) unit.



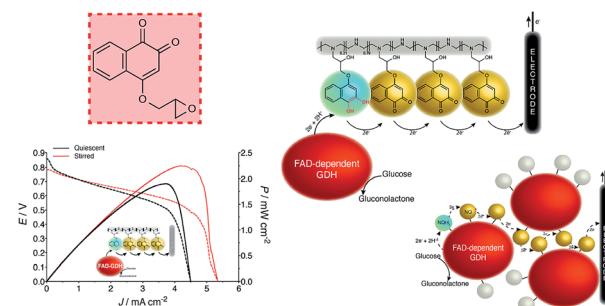
## EDGE ARTICLES

4867

**Rational design of quinones for high power density biofuel cells**

Ross D. Milton, David P. Hickey, Sofiene Abdellaoui, Koun Lim, Fei Wu, Boxuan Tan and Shelley D. Minteer\*

Rationally designing quinones to label GDH and create a redox hydrogel that delivers high OCP, current and power densities.

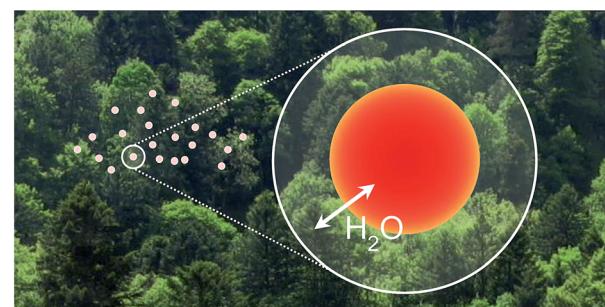


4876

**Water diffusion in atmospherically relevant  $\alpha$ -pinene secondary organic material**

Hannah C. Price,\* Johan Mattsson, Yue Zhang, Allan K. Bertram, James F. Davies, James W. Grayson, Scot T. Martin, Daniel O'Sullivan, Jonathan P. Reid, Andrew M. J. Rickards and Benjamin J. Murray\*

We report the first direct measurements of water diffusion coefficients in secondary organic aerosol.

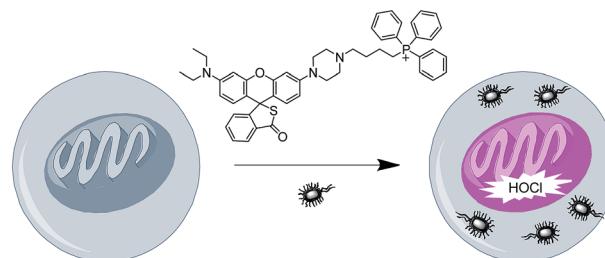


4884

**HOCl can appear in the mitochondria of macrophages during bacterial infection as revealed by a sensitive mitochondrial-targeting fluorescent probe**

Jin Zhou, Lihong Li, Wen Shi,\* Xinghui Gao, Xiaohua Li and Huimin Ma\*

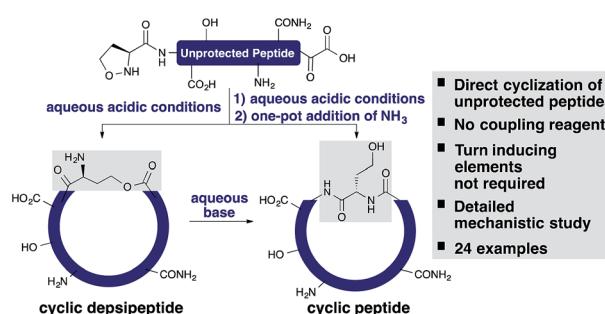
HOCl can appear in the mitochondria of macrophages during bacterial infection as revealed by a new sensitive mitochondrial-targeting fluorescent probe.



4889

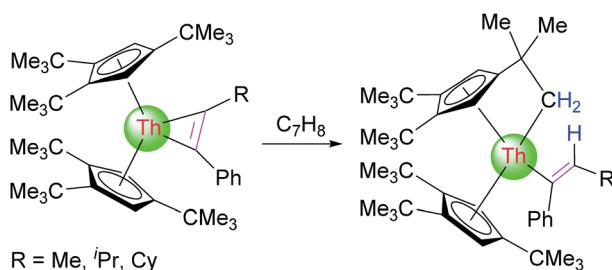
**Spontaneous head-to-tail cyclization of unprotected linear peptides with the KAHA ligation**

Florian Rohrbacher, Gildas Deniau, Anatol Luther and Jeffrey W. Bode\*

The  $\alpha$ -ketoacid–hydroxylamine (KAHA) ligation enables the direct cyclization of unprotected peptides upon cleavage, without coupling reagents or purification of precursors. We report the synthesis of a library of 24 cyclic peptides and a detailed mechanistic study.

## EDGE ARTICLES

4897

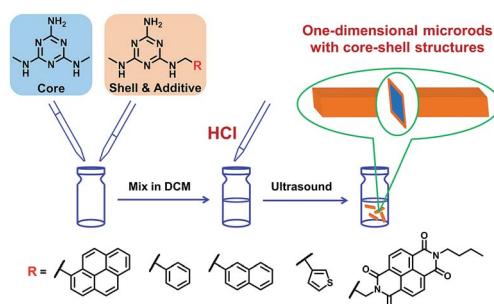


**C–H bond activation induced by thorium metallacycloprenes: a combined experimental and computational study**

Bo Fang, Lei Zhang, Guohua Hou, Guofu Zi,\* De-Cai Fang\* and Marc D. Walter\*

Thorium metallacycloprenes derived from phenyl(alkyl) acetylenes are very reactive complexes that undergo selective intramolecular C–H bond activation.

4907

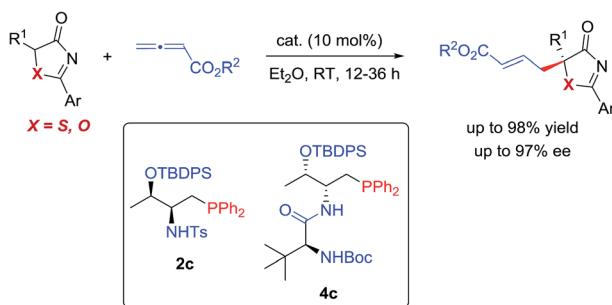


**Self-assembling 1D core/shell microrods by the introduction of additives: a one-pot and shell-tunable method**

Jun Xu, Hongde Yu, Liulin Yang, Guanglu Wu, Zhiqiang Wang, Dong Wang and Xi Zhang\*

A one-pot method for the fabrication of 1D core/shell microrods with tunable shell compositions by the introduction of additives.

4912

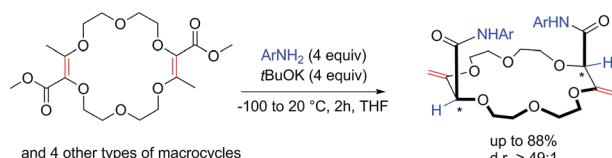


**Highly enantioselective construction of tertiary thioethers and alcohols *via* phosphine-catalyzed asymmetric  $\gamma$ -addition reactions of 5H-thiazol-4-ones and 5H-oxazol-4-ones: scope and mechanistic understandings**

Tianli Wang, Zhaoyuan Yu, Ding Long Hoon, Kuo-Wei Huang, Yu Lan\* and Yixin Lu\*

A new method for facile access to enantioenriched tertiary thioethers/alcohols.

4923



**Remote stereoselective deconjugation of  $\alpha,\beta$ -unsaturated esters by simple amidation reactions**

Mahesh Vishe, Radim Hrdina, Amalia I. Poblador-Bahamonde, Céline Besnard, Laure Guénée, Thomas Bürgi and Jérôme Lacour\*

The amidation of macrocyclic conjugated esters affords in one-pot single (chiral)  $\beta,\gamma$ -unsaturated diastereomers *via* effective remote stereocontrol.



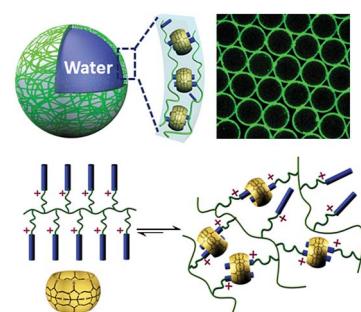
## EDGE ARTICLES

4929

**Supramolecular hydrogel microcapsules via cucurbit[8]uril host–guest interactions with triggered and UV-controlled molecular permeability**

Ziyi Yu, Jing Zhang, Roger J. Coulston, Richard M. Parker, Frank Biedermann, Xin Liu, Oren A. Scherman\* and Chris Abell\*

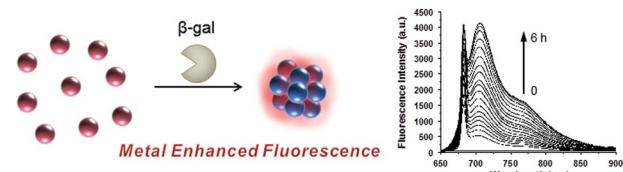
Host–guest assembly at the interface of microfluidic droplets offers a versatile strategy to construct supramolecular hydrogel microcapsules with “smart” cargo release.



4934

**An enzyme-responsive metal-enhanced near-infrared fluorescence sensor based on functionalized gold nanoparticles**

Zhanghua Zeng, Shin Mizukami, Katsumasa Fujita and Kazuya Kikuchi\*

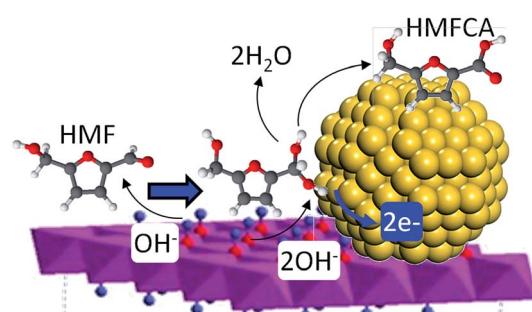
An enzyme-responsive NIR nanosystem based on MEF was fabricated by surface functionalization of gold nanoparticles. Sensors based on this strategy are promising for enzyme detection in early diagnostic imaging and *in vivo* applications.

4940

**Solid base catalysed 5-HMF oxidation to 2,5-FDCA over Au/hydrotalcites: fact or fiction?**

Leandro Ardeman, Giannantonio Cibin, Andrew J. Dent, Mark A. Isaacs, Georgios Kyriakou, Adam F. Lee,\* Christopher M. A. Parlett, Stephen A. Parry and Karen Wilson\*

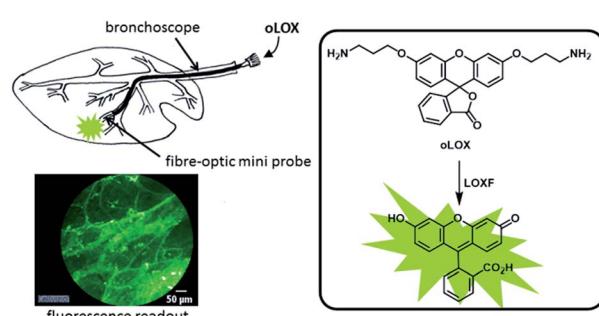
Synergistic effects between alkali-free hydrotalcites and gold nanoparticles afford efficient heterogeneous catalysts for the cascade oxidation of 5-HMF to 2,5-FDCA.



4946

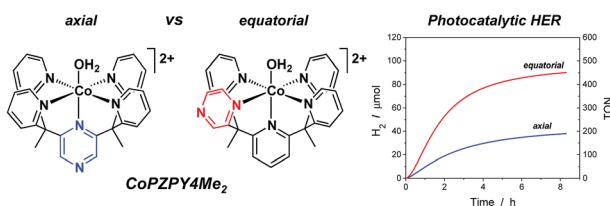
**Optical molecular imaging of lysyl oxidase activity – detection of active fibrogenesis in human lung tissue**

Tashfeen Aslam, Amy Miele, Sunay V. Chankeshwara, Alicia Megia-Fernandez, Chesney Michels, Ahsan R. Akram, Neil McDonald, Nik Hirani, Chris Haslett, Mark Bradley\* and Kevin Dhalialiwal\*

A fluorogenic probe provides real-time measurement of lysyl oxidase activity in *ex vivo* asinine and human lung tissue.

## EDGE ARTICLES

4954

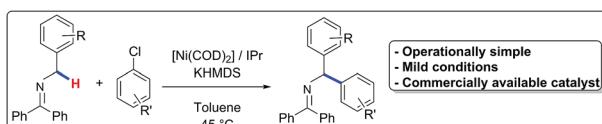


**Bioinspired design of redox-active ligands for multielectron catalysis: effects of positioning pyrazine reservoirs on cobalt for electro- and photocatalytic generation of hydrogen from water**

Jonah W. Jurss, Rony S. Khnayzer, Julien A. Panetier, Karim A. El Roz, Eva M. Nichols, Martin Head-Gordon,\* Jeffrey R. Long,\* Felix N. Castellano\* and Christopher J. Chang\*

We report the effects of installing redox-active pyrazines at distinct positions in a series of isostructural Co catalysts.

4973

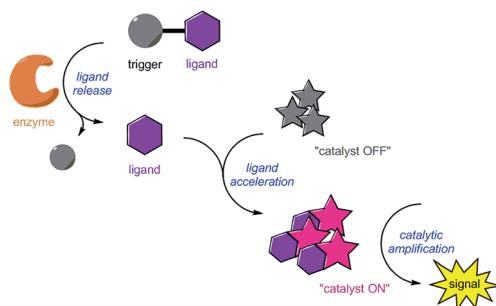


**Synthesis of ( diarylmethyl)amines using Ni-catalyzed arylation of C(sp<sup>3</sup>)-H bonds**

José A. Fernández-Salas, Enrico Marelli and Steven P. Nolan\*

The first nickel catalyzed deprotonative cross coupling between C(sp<sup>3</sup>)-H bonds and aryl chlorides is reported, allowing the challenging arylation of benzylimines in the absence of directing group or stoichiometric metal activation.

4978

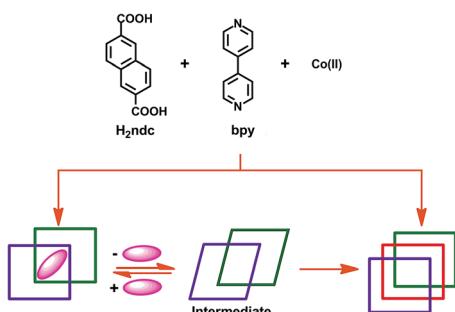


**Signal transduction and amplification through enzyme-triggered ligand release and accelerated catalysis**

Sean Goggins, Barrie J. Marsh, Anneke T. Lubben and Christopher G. Frost\*

An enzyme-triggered catalytic signal amplification cascade is described through the design of a novel enzyme substrate that selectively activates an organometallic transfer hydrogenation catalyst once triggered.

4986



**Isolation of a structural intermediate during switching of degree of interpenetration in a metal-organic framework**

Himanshu Aggarwal, Raj Kumar Das, Prashant M. Bhatt and Leonard J. Barbour\*

A structural intermediate has been isolated for the first time during switching of interpenetration from twofold to threefold in the MOF [Co<sub>2</sub>(ndc)<sub>2</sub>(bpy)].

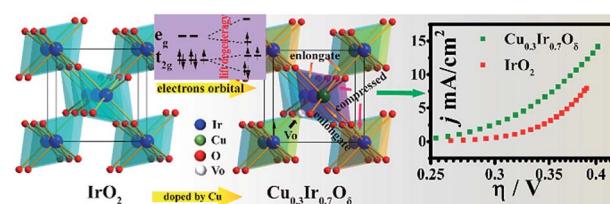


## EDGE ARTICLES

4993

**An efficiently tuned d-orbital occupation of  $\text{IrO}_2$  by doping with Cu for enhancing the oxygen evolution reaction activity**

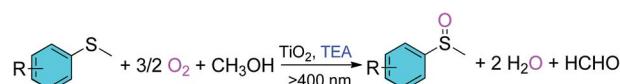
Wei Sun, Ya Song, Xue-Qing Gong,\* Li-mei Cao and Ji Yang\*

Tuning Ir d-orbital occupation via doping Cu into the  $\text{IrO}_2$  lattice to prepare a highly efficient oxygen evolution reaction catalyst,  $\text{Cu}_{0.3}\text{Ir}_{0.7}\text{O}_\delta$ .

5000

**Tertiary amine mediated aerobic oxidation of sulfides into sulfoxides by visible-light photoredox catalysis on  $\text{TiO}_2$** 

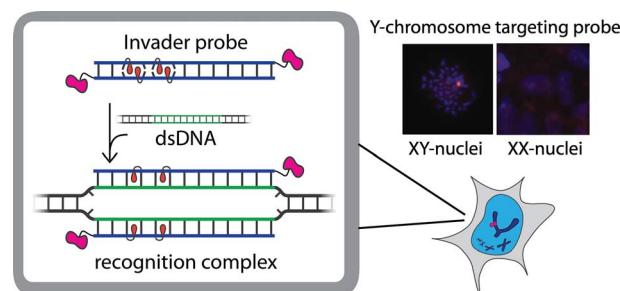
Xianjun Lang, Wei Hao, Wan Ru Leow, Shuzhou Li,\* Jincai Zhao and Xiaodong Chen\*

The selective aerobic oxidation of sulfides into sulfoxides on  $\text{TiO}_2$  under visible-light irradiation was accomplished through synergistic catalysis with triethylamine.

5006

**Invader probes: harnessing the energy of intercalation to facilitate recognition of chromosomal DNA for diagnostic applications**

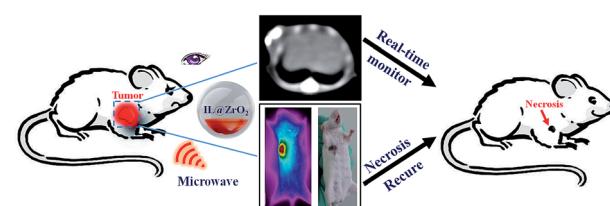
Dale C. Guenther, Grace H. Anderson, Saswata Karmakar, Brooke A. Anderson, Bradley A. Didion, Wei Guo, John P. Verstegen and Patrick J. Hrdlicka\*

Optimized Invader probes enable efficient ( $C_{50} < 1 \mu\text{M}$ ), fast ( $t_{50} < 3 \text{ h}$ ), kinetically stable ( $>24 \text{ h}$ ), and single nucleotide specific recognition of DNA targets.

5016

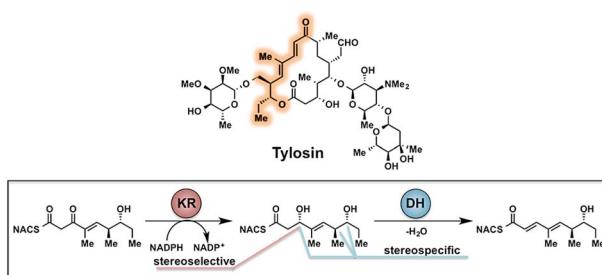
**A smart all-in-one theranostic platform for CT imaging guided tumor microwave thermotherapy based on  $\text{IL@ZrO}_2$  nanoparticles**

Haitang Shi, Meng Niu, Longfei Tan, Tianlong Liu, Haibo Shao, Changhui Fu, Xiangling Ren, Tengchuang Ma, Jun Ren, Linlin Li, Huiyu Liu, Ke Xu,\* Jianxin Wang, Fangqiong Tang and Xianwei Meng\*

This paper develops a simple multifunctional theranostic platform using an  $\text{IL@ZrO}_2$  nanostructure for CT imaging guided tumor microwave thermotherapy.

## EDGE ARTICLES

5027

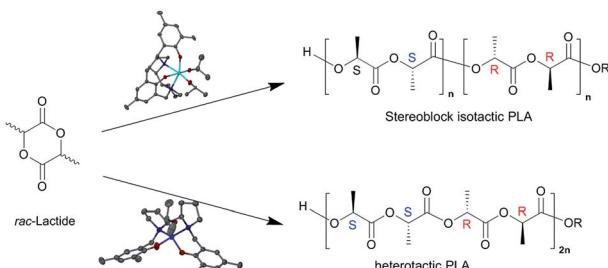


**Tylosin polyketide synthase module 3: stereospecificity, stereoselectivity and steady-state kinetic analysis of  $\beta$ -processing domains *via* diffusible, synthetic substrates**

William D. Fiers, Greg J. Dodge, Yang Li, Janet L. Smith, Robert A. Fecik\* and Courtney C. Aldrich\*

Natural and modified substrates coupled with LC-MS/MS analysis of products revealed the stereospecificity and stereoselectivity of a polyketide didomain.

5034

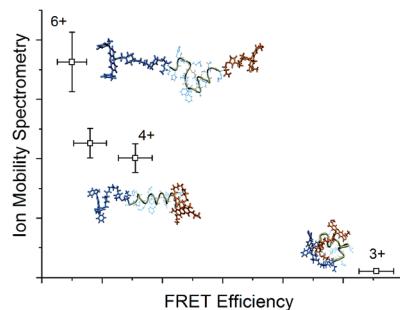


**Metal influence on the iso- and hetero-selectivity of complexes of bipyrrrolidine derived salan ligands for the polymerisation of rac-lactide**

Matthew D. Jones,\* Lauren Brady, Paul McKeown, Antoine Buchard, Pascal M. Schäfer, Lynne H. Thomas, Mary F. Mahon, Timothy J. Woodman and John P. Lowe

A series of complexes based on 2,2'-bipyrrrolidine based salan ligands have been prepared and either isotactic or heterotactic PLA have been prepared.

5040

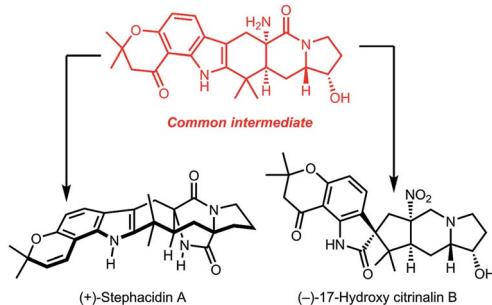


**Conformational changes in amyloid-beta (12–28) alloforms studied using action-FRET, IMS and molecular dynamics simulations**

Steven Daly, Alexander Kulesza, Frederic Poussigue, Anne-Laure Simon, Chang Min Choi, Geoffrey Knight, Fabien Chirot, Luke MacAleece, Rodolphe Antoine and Philippe Dugourd\*

The gas phase conformations of two amyloid beta mutants are studied by multiple techniques to elucidate the origin of the different aggregation behaviour.

5048



**Unified approach to prenylated indole alkaloids: total syntheses of (–)-17-hydroxy-citrinalin B, (+)-stephacidin A, and (+)-notoamide I**

Eduardo V. Mercado-Marin and Richmond Sarpong\*

The first strategy that provides reverse-prenylated indole alkaloids that bear a characteristic bicyclo[2.2.2]diazaoctane as well as those that lack this structural motif is reported.



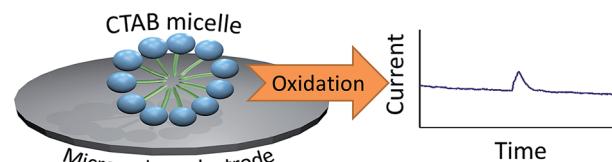
## EDGE ARTICLES

5053

**Electrochemical detection of single micelles through 'nano-impacts'**

H. S. Toh and R. G. Compton\*

CTAB (cetyltrimethylammonium bromide) micelles are detected directly *via* the novel electrochemical method of 'nano-impacts' through oxidation of its bromide content.

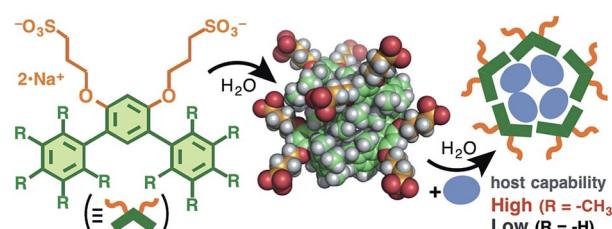


5059

**Well-defined aqueous nanoassemblies from amphiphilic *meta*-terphenyls and their guest incorporation**

Yusuke Okazawa, Kei Kondo, Munetaka Akita and Michito Yoshizawa\*

Spherical molecular assemblies with diameters of  $\sim 2$  nm were quantitatively formed in water from new amphiphilic *meta*-terphenyls and the nanoassembly with methyl groups provides superior host capability for fluorescent dyes.



5063

**Site-selective formation of an iron(IV)-oxo species at the more electron-rich iron atom of heteroleptic  $\mu$ -nitrido diiron phthalocyanines**

Ümit İşçi, Abayomi S. Faponle, Pavel Afanasiev, Florian Albrieux, Valérie Briois, Vefa Ahsen, Fabienne Dumoulin,\* Alexander B. Sorokin\* and Sam P. de Visser\*

A combination of MS and computation on  $\mu$ -nitrido bridged diiron complexes reveals  $\text{H}_2\text{O}_2$  binding to the complex and generates an oxidant capable of oxidizing methane.

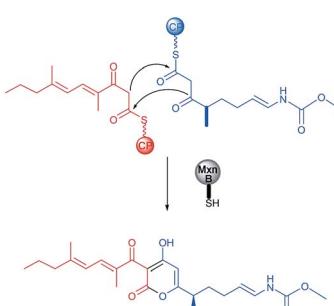


5076

***In vitro* reconstitution of  $\alpha$ -pyrone ring formation in myxopyronin biosynthesis**

H. Sucipto, J. H. Sahner, E. Prusov, S. C. Wenzel, R. W. Hartmann, J. Koehnke\* and R. Müller\*

$\alpha$ -Pyrone rings exist in many polyketide synthase (PKS) derived natural products. We report the first *in vitro* reconstitution of  $\alpha$ -pyrone ring formation by a type I PKS using chemically synthesized substrates.



## CORRECTIONS

5086

**Correction: Pattern-based detection of anion pollutants in water with DNA polyfluorophores**

Hyukin Kwon, Wei Jiang and Eric T. Kool\*

5087

**Correction: Assessing the exchange coupling in binuclear lanthanide(III) complexes and the slow relaxation of the magnetization in the antiferromagnetically coupled Dy<sub>2</sub> derivative**

Chun Y. Chow, Hélène Bolvin, Victoria E. Campbell, Régis Guillot, Jeff W. Kampf, Wolfgang Wernsdorfer, Frédéric Gendron, Jochen Autschbach, Vincent L. Pecoraro\* and Talal Mallah\*

5088

**Correction: A supramolecular strategy for tuning the energy level of naphthalenediimide: promoted formation of radical anions with extraordinary stability**

Qiao Song, Fei Li, Zhiqiang Wang and Xi Zhang\*

5090

**Correction: Supramolecularly engineered phospholipids constructed by nucleobase molecular recognition: upgraded generation of phospholipids for drug delivery**

Dali Wang, Chunlai Tu, Yue Su, Chuan Zhang, Udo Greiser, Xinyuan Zhu,\* Deyue Yan and Wenxin Wang\*