

# Organic & Biomolecular Chemistry

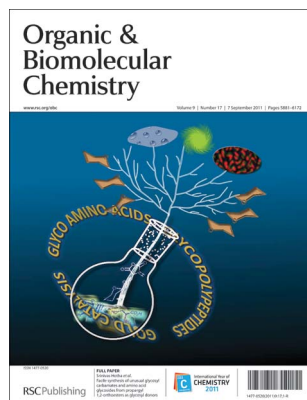
An international journal of synthetic, physical and biomolecular organic chemistry

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## IN THIS ISSUE

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### Cover

See Ashif Y. Shaikh *et al.*,  
pp. 5951–5959.

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*Biomol. Chem.*, 2011, **9**, 5951.

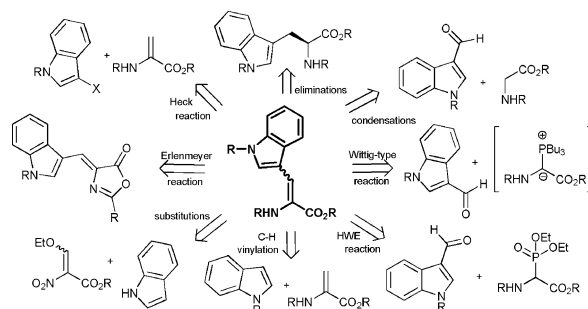
## PERSPECTIVES

5897

### The synthesis of dehydrotryptophan and dehydrotryptophan-containing peptides

Harveen Kaur, Amanda M. Heapy and Margaret A. Brimble\*

The synthesis of dehydrotryptophan and its derivatives is summarised. The occurrence of dehydrotryptophan in peptide based natural products is driving the need for its general use as a building block for peptide synthesis.

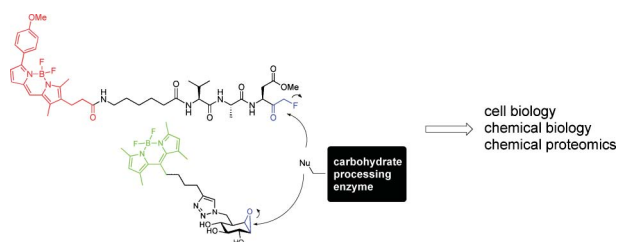


5908

### Irreversible inhibitors and activity-based probes as research tools in chemical glycobiology

Martin D. Witte, Gijsbert A. van der Marel, Johannes M. F. G. Aerts and Herman S. Overkleeft\*

This review gives an overview of the enzymes involved in the degradation of glycoconjugates, and the activity-based probes that have been applied to study these enzymes and the (dis)advantages of these probes.



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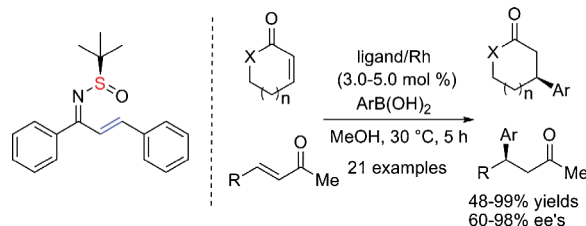
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5927

### Chiral *N*-*tert*-butanesulfinyl $\alpha,\beta$ -unsaturated ketimine: a simple and highly effective olefin/sulfinimide hybrid ligand for asymmetric 1,4-additions

Xiangqing Feng, Beibei Wei, Jing Yang\* and Haifeng Du\*

Chiral olefin/sulfinimide hybrid ligands prepared through a one-step condensation of  $\alpha,\beta$ -unsaturated ketones with *tert*-butanesulfinamide were found to be highly effective for rhodium-catalyzed asymmetric 1,4-additions.

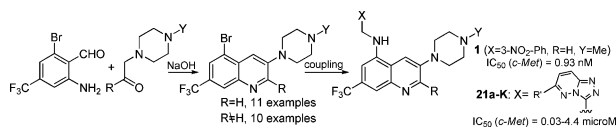


5930

### Expedient one-pot synthesis of C3-piperazinyl-substituted quinolines: key precursors to potent c-Met inhibitors

Yuanxiang Wang, Jing Ai, Gang Liu, Meiyu Geng\* and Ao Zhang\*

An effective one-pot synthesis of quinolines bearing diverse C3-piperazinyl functions was developed. This method not only enables the synthesis of our early reported c-Met inhibitor in large scale, but also provides a way to generate novel multi-substituted quinolines for further structure-activity relationship (SAR) study.

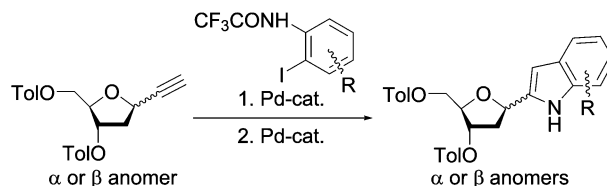


5934

### Modular synthesis of 1- $\alpha$ - and 1- $\beta$ -(indol-2-yl)-2'-deoxyribose C-nucleosides

David Nečas, Denisa Hidasová, Michal Hocek\* and Martin Kotora\*

The synthesis is based on the Sonogashira reaction of 1 $\alpha$ - and 1 $\beta$ -ethynyldeoxyribose and 2-haloanilines followed by a Pd-complex catalyzed cyclization.

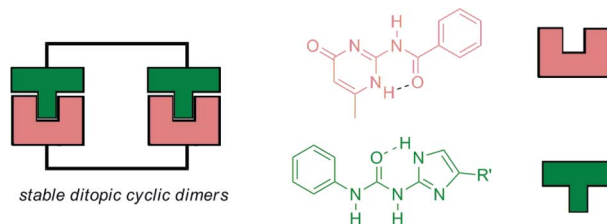


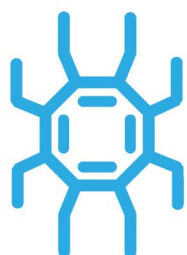
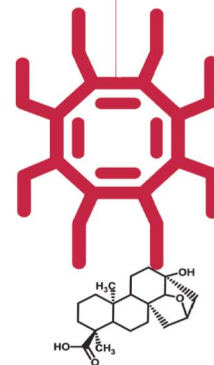
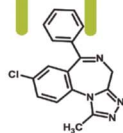
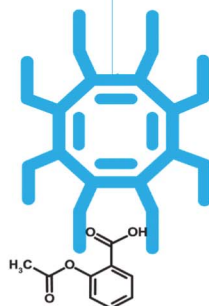
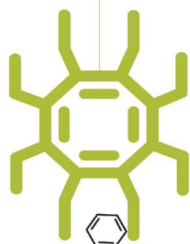
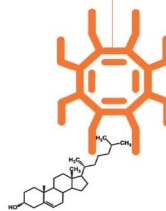
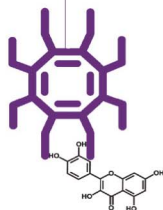
5938

### Ditopic triply hydrogen-bonded heterodimers

Adam Gooch, Simon Barrett, Julie Fisher, Christopher I. Lindsay and Andrew J. Wilson\*

This paper describes the synthesis and self-assembly of a stable hydrogen-bonded heterodimer comprising ditopic ureidoimidazole and amidoisocytosine motifs.





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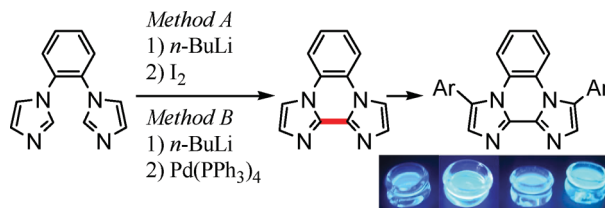
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5941

### Novel formation of diimidazo[1,2-*a*:2',1'-*c*]quinoxaline derivatives and their optical properties

Shoji Matsumoto,\* Erdenebolor Batmunkh, Motohiro Akazome, Yoshiyuki Takata and Michiko Tamano

Reaction of 1,2-di(imidazolyl)benzene gave diimidazo[1,2-*a*:2',1'-*c*]quinoxaline, whose 3,10-diarylated derivatives emits blue fluorescence.

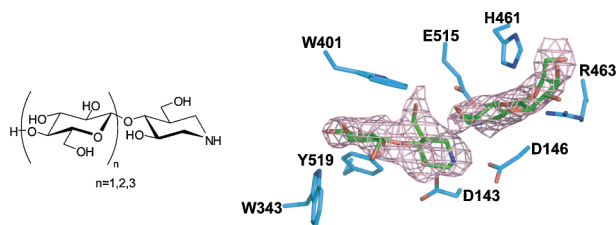


5945

### A potential fortuitous binding of inhibitors of an inverting family GH9 $\beta$ -glycosidase derived from isofagomine

Solange Moréra,\* Armelle Vigouroux and Keith A. Stubbs\*

Using structural insight, the binding mode of isofagomine-derived inhibitors with a family GH9 endoglucanase from *Alicyclobacillus acidocaldarius* (*AaCel9A*) reveals a novel binding mode for these compounds compared to inverting glycosidases from family GH6.

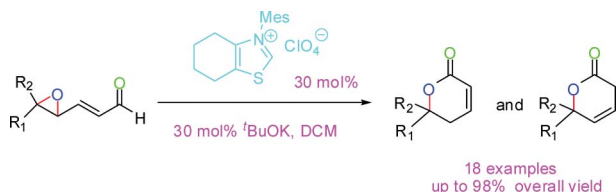


5948

### *N*-Heterocyclic carbene-catalyzed cascade epoxide-opening and lactonization reaction for the synthesis of dihydropyrone derivatives

Jing Qi, Xingang Xie, Jinmei He, Ling Zhang, Donghui Ma and Xuegang She\*

*N*-Heterocyclic carbene was employed as an efficient organic catalyst to catalyze a cascade epoxide-opening and lactonization reaction. This organocatalytic process could transform various readily accessible  $\gamma$ -epoxy- $\alpha,\beta$ -enals into dihydropyrone derivatives in good to excellent yields.



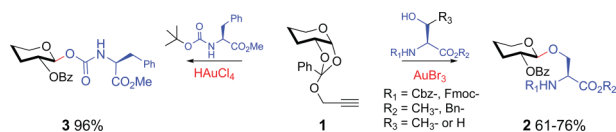
## PAPERS

5951

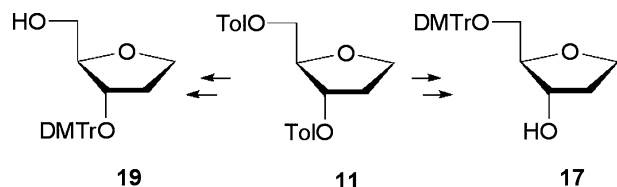
### Facile synthesis of unusual glycosyl carbamates and amino acid glycosides from propargyl 1,2-orthoesters as glycosyl donors

Ashif Y. Shaikh, Gopalsamy Sureshkumar, Debasish Pati, Sayam Sen Gupta and Srinivas Hotha\*

Amino acid glycoconjugates (O-glycosides **2** or glycosyl urethanes **3**) were synthesized in very high yields exploiting salient features of gold catalyzed glycosidations.



5960



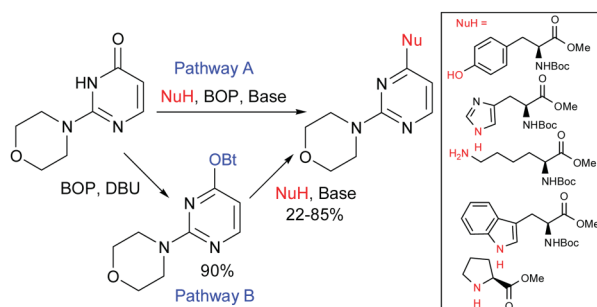
## Chemoenzymatic procedures

## An expedient biocatalytic procedure for abasic site precursors useful in oligonucleotide synthesis

Sául Martínez-Montero, Susana Fernández, Yogesh S. Sanghvi, Vicente Gotor\* and Miguel Ferrero\*

A fast, atom-efficient and convenient synthesis of 3-, and 5-*O*-DMTr-1,2-dideoxy-D-ribose **17** and **19** has been achieved, yielding a practical and scalable green method useful for industrial applications.

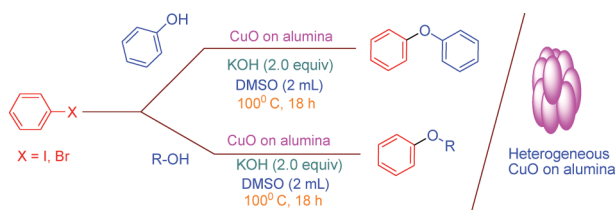
5967

Coupling reaction between electron-rich pyrimidinones and  $\alpha$ -amino acids promoted by phosphonium salts

Abdelatif ElMarrouni, Josep M. Fabrellas and Montserrat Heras\*

First coupling reaction between an electron-rich 2-morpholino-4(3*H*)-pyrimidinone and nucleophilic side chains of several natural  $\alpha$ -amino acids promoted by phosphonium salts has been achieved.

5978

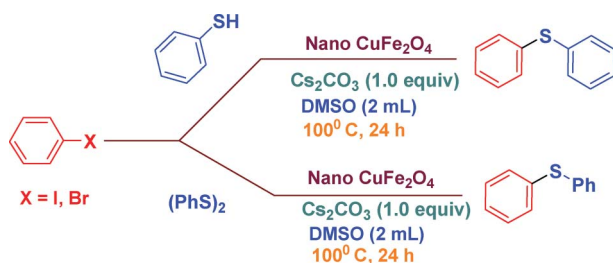


## Recyclable heterogeneous copper oxide on alumina catalyzed coupling of phenols and alcohols with aryl halides under ligand-free conditions

Kokkerala Swapna, Sabbavarapu Narayana Murthy, Mocharla Tarani Jyothi and Yadavalli Venkata Durga Nageswar\*

A simple and efficient alumina-supported CuO-catalyzed *O*-arylation of phenols and aliphatic alcohols with various aryl as well as heteroaryl halides under ligand-free conditions are reported. The explored catalyst is inexpensive, air-stable and recyclable.

5989

Nano-CuFe<sub>2</sub>O<sub>4</sub> as a magnetically separable and reusable catalyst for the synthesis of diaryl/aryl alkyl sulfides via cross-coupling process under ligand-free conditions

Kokkerala Swapna, Sabbavarapu Narayana Murthy, Mocharla Tarani Jyothi and Yadavalli Venkata Durga Nageswar\*

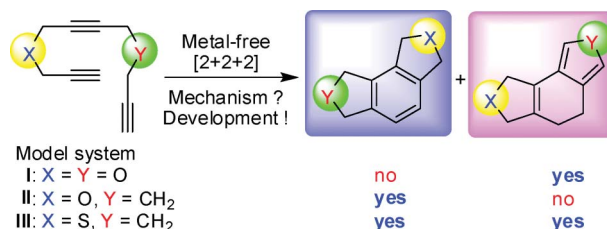
A simple and efficient nano CuFe<sub>2</sub>O<sub>4</sub> catalyzed *S*-arylation of thiols and disulfides with various aryl, aliphatic as well as heteroaryl halides under ligand-free conditions are reported. The catalyst is air-stable, inexpensive, magnetically separable and recyclable up to four cycles.

5997

### Theoretical insights into the metal-free and formal [2 + 2 + 2] cycloaddition strategy *via* intramolecular cascade propargylic ene/Diels–Alder reactions with tautomerization

Xinyao Li and Jiayi Xu\*

The [2 + 2 + 2] cycloadditions show different regioselectivities and types of final products due to different tethered atoms between the yne moieties.

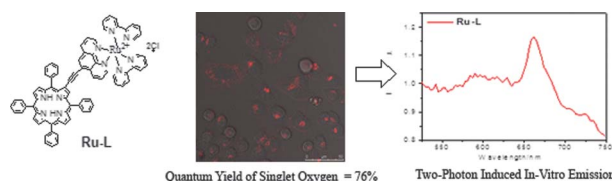


6004

### Two-photon induced luminescence, singlet oxygen generation, cellular uptake and photocytotoxic properties of amphiphilic Ru(II) polypyridyl–porphyrin conjugates as potential bifunctional photodynamic therapeutic agents

JingXiang Zhang, Ka-Leung Wong, Wai-Kwok Wong,\* Nai-Ki Mak, Daniel W. J. Kwong and Hoi-Lam Tam

Ru(II) polypyridyl-porphyrin(Ru-L) can be excited and emits in the “biological window” *in vitro*, making it a potential potent new generation photodynamic therapeutic agent capable of singlet oxygen generation and *in vitro* near-infrared emission.

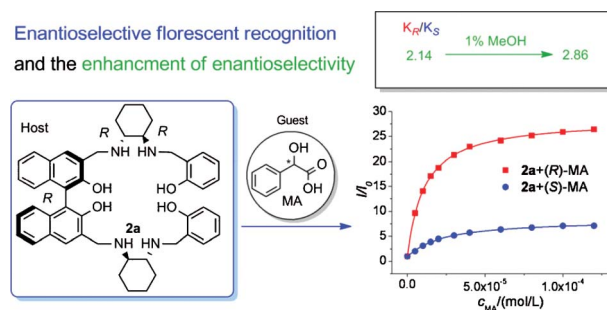


6011

### Enantioselective fluorescent recognition of mandelic acid by unsymmetrical salalen and salan sensors

Xia Yang, Xuechao Liu, Kang Shen, Yong Fu, Ming Zhang, Chengjian Zhu\* and Yixiang Cheng\*

Unsymmetrical salan sensor towards mandelic acids exhibits highly fluorescent sensitivity and enantioselectivity which can be higher in mixed solvent.

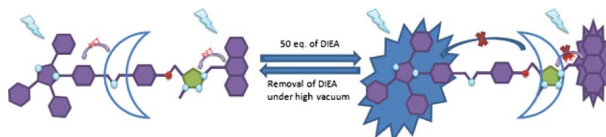


6022

### Synthesis of a [2]rotaxane operated in basic environment

Wenlong Yang, Yongjun Li,\* Jianhong Zhang, Yanwen Yu, Taifeng Liu, Huibiao Liu and Yuliang Li\*

A tight [2]rotaxane with two chromophores as stoppers is described. The macrocycle can reversibly move under basic conditions which can lead to changes in the photo-physical properties of the supramolecular system.



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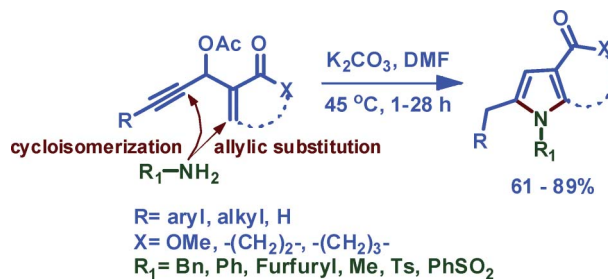


6027

### Morita–Baylis–Hillman acetates of acetylenic aldehydes: versatile synthons for substituted pyrroles *via* a metal-free tandem reaction

Chada Raji Reddy,\* Motatipally Damoder Reddy, Boinapally Srikanth and Kothakonda Rajendra Prasad

A simple and novel method for one-pot synthesis of substituted pyrroles starting from MBH acetates of acetylenic aldehydes with amines using a tandem reaction involving allylic substitution/cycloisomerization is described.

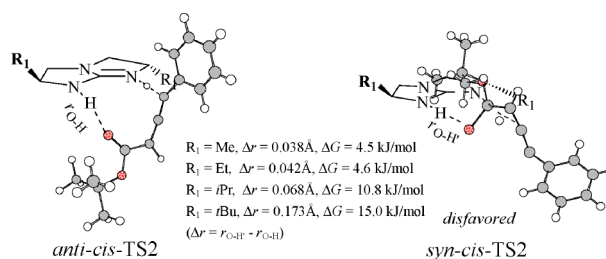


6034

### Computational investigation on the mechanism and stereochemistry of guanidine-catalyzed enantioselective isomerization of 3-alkynoates to allenates

Dongfeng Huang, Song Qin\* and Changwei Hu\*

Larger R<sub>1</sub> groups could selectively make one of the competing transition states unstable by decreasing the strength of the N–H···O hydrogen-bond.

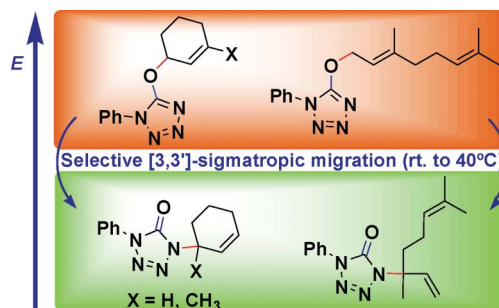


6040

### Sigmatropic rearrangements in 5-allyloxytetrazoles

Luís M. T. Frija, Igor Reva,\* Amin Ismael, Daniela V. Coelho, Rui Fausto and M. Lurdes S. Cristiano\*

Thermal isomerization of allyl tetrazolyl ethers derived from cyclohex-2-enol (X = H), 3-methylcyclohex-2-enol (X = CH<sub>3</sub>), and from nerol was found to occur exclusively through a [3,3']-sigmatropic migration of the allylic system from *O* to *N*. The process is kinetically controlled.

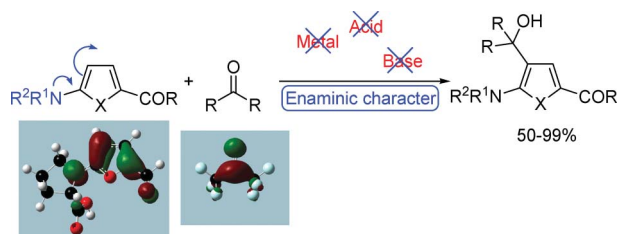


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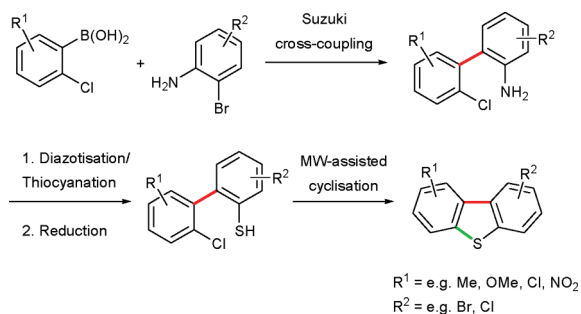
### A facile preparation of trisubstituted amino-furan and -thiophene derivatives

Raouf Medimagh, Sylvain Marque,\* Damien Prim\* and Saber Chatti

The ability of both heterocycles to undergo selective  $\beta$ -alkylation is compared by mean of experimental and theoretical data.



6066

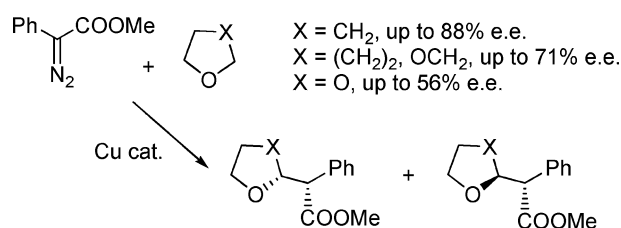


### Versatile synthesis of functionalised dibenzothiophenes via Suzuki coupling and microwave-assisted ring closure

Sonsoles Rodriguez-Aristegui, Kate M. Clapham,\* Lauren Barrett, Céline Cano, Marine Desage-El Murr, Roger J. Griffin, Ian R. Hardcastle, Sara L. Payne, Tommy Rennison, Caroline Richardson and Bernard T. Golding\*

Suzuki coupling of 2,6-dibromoaniline with a phenylboronic acid aided by microwave irradiation gave amino-biphenyls; conversion of amino into thiol followed by base-induced intramolecular substitution, generated dibenzothiophenes.

6075

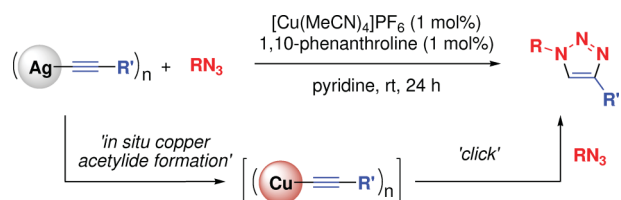


### Enantioselective C–H carbene insertions with homogeneous and immobilized copper complexes

José M. Fraile,\* Pilar López-Ram-de-Viu, José A. Mayoral, Marta Roldán and Jorge Santafé-Valero

Immobilization of chiral copper complexes allows the enantioselective carbene insertion into C–H bonds of poorly reactive cyclic ethers.

6082

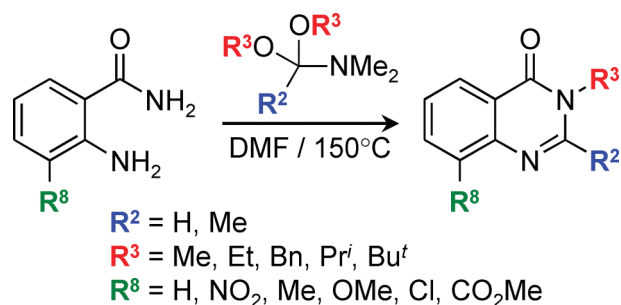


### Copper(I)-catalyzed cycloaddition of silver acetylides and azides: Incorporation of volatile acetylenes into the triazole core

Ilaria Proietti Silvestri, Fikre Andemarian, George N. Khairallah, Su Wan Yap, Tim Quach, Sammi Tsegay, Craig M. Williams, Richard A. J. O'Hair, Paul S. Donnelly and Spencer J. Williams\*

Copper(I)-catalyzes the cycloaddition of silver acetylides and azides to afford 1,4-disubstituted triazoles via *in situ* transmetalation to copper acetylides.

6089



### *N*<sup>3</sup>-Alkylation during formation of quinazolin-4-ones from condensation of anthranilamides and orthoamides

Amit Nathubhai, Richard Patterson, Timothy J. Woodman, Harriet E. C. Sharp, Miranda T. Y. Chui, Hugo H. K. Chung, Stephanie W. S. Lau, Jun Zheng, Matthew D. Lloyd, Andrew S. Thompson and Michael D. Threadgill\*

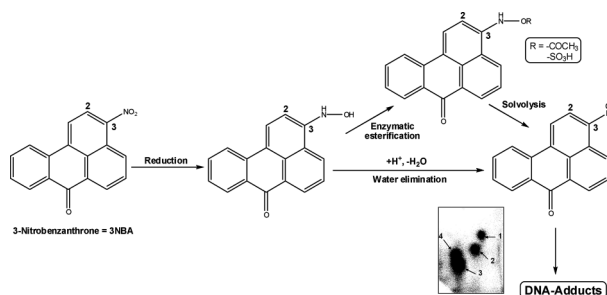
Reaction of anthranilamides with orthoamides ( $R^3 = \text{primary alkyl}$ ) gives *N*<sup>3</sup>-alkylated quinazolin-4-ones; when  $R^3 = \text{Pr}^t$ , *N*<sup>3</sup>-methylation competes, indicating that the intermediate alkoxyiminium cation has three potential electrophilic sites.

6100

### Theoretical investigations on the formation of nitrobenzanthrone-DNA Adducts

Volker M. Arlt, David H. Phillips and Jóhannes Reynisson\*

Metabolic activation of 3-NBA leading to the formation of its arylnitrenium ion and consequent DNA adduct formation. The thermochemical formation cascades were calculated for six 3-NBA-derived DNA adducts using DFT. Clear exothermic pathways were found for four adducts and all have been observed to be formed in experimental systems.

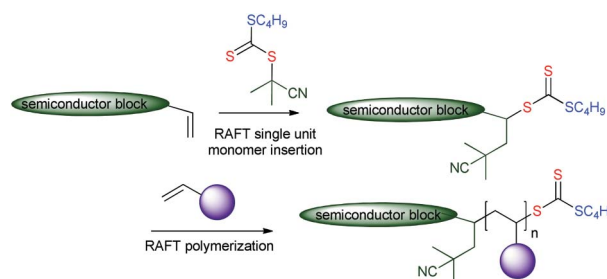


6111

### Block copolymers containing organic semiconductor segments by RAFT polymerization

Ming Chen,\* Matthias Häussler,\* Graeme Moad\* and Ezio Rizzardo

Vinyl compounds can be transformed to macro-RAFT agents by RAFT single unit monomer insertion providing a route to block copolymers with a short, non-hydrolysable block linkage.

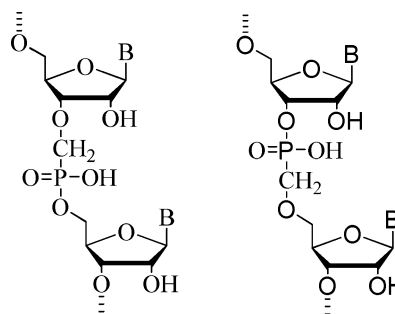


6120

### Synthesis of oligoribonucleotides with phosphonate-modified linkages

Ondřej Páv, Ivana Košiová, Ivan Barvík, Radek Pohl, Miloš Buděšínský and Ivan Rosenberg\*

Solid phase synthesis of phosphonate oligoribonucleotides using 2'-O-benzoyloxymethoxymethyl protected monomers is presented in both 3'→5' and 5'→3' directions.

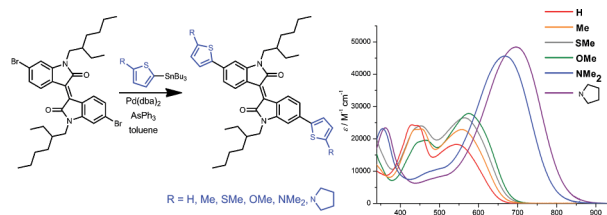


6127

### Thiophene-functionalized isoindigo dyes bearing electron donor substituents with absorptions approaching the near infrared region

David Bialas, Sabin-Lucian Suraru, Ralf Schmidt and Frank Würthner\*

Synthesis and electronic properties of a series of thiophene-functionalized isoindigo derivatives with strong absorptions reaching the NIR region are reported.





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Registration deadline 25 May 2012  
[www.rsc.org/FD157](http://www.rsc.org/FD157)

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Poster abstracts by 11 May 2012  
Registration deadline 8 June 2012  
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Registration deadline 3 August 2012  
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Registration deadline 3 August 2012  
[www.rsc.org/ICPOC21](http://www.rsc.org/ICPOC21)

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10–12 September 2012, Sheffield, UK  
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[www.rsc.org/FD161](http://www.rsc.org/FD161)

## Lipids and Membrane Biophysics (Faraday Discussion 161)

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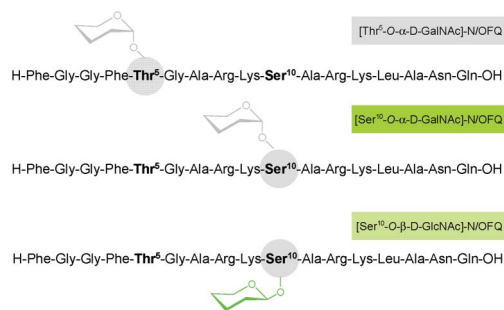
More 2012 events will be announced soon - check the website for details

6133

### Synthesis, biological evaluation and structural characterization of novel glycopeptide analogues of nociceptin N/O/FQ

Gemma Arsequell, Mònica Rosa, Carlos Mayato, Rosa L. Dorta, Verónica Gonzalez-Nunez, Katherine Barreto-Valer, Filipa Marcelo, Luis P. Calle, Jesús T. Vázquez, Raquel E. Rodríguez, Jesús Jiménez-Barbero and Gregorio Valencia\*

Folded hairpin-like conformations seem to be the cause of the inactivity of one of the nociceptin glycopeptides studied.

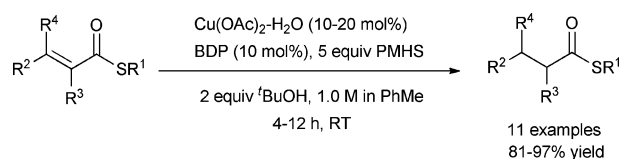


6143

### Conjugate reduction and reductive aldol cyclization of $\alpha,\beta$ -unsaturated thioesters catalyzed by (BDP)CuH

Ninglin Li, Jun Ou, Michel Miesch and Pauline Chiu\*

A range of  $\alpha,\beta$ -unsaturated thioesters undergo conjugate reductions effectively with PMHS, catalyzed by  $\text{Cu}(\text{OAc})_2\text{-H}_2\text{O}$  with BDP as ligand.

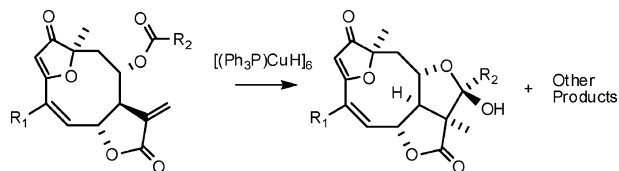


6148

### Selectivity in reduction of natural furanoheliangolides with Stryker's reagent

Daiane C. Sass, Vladimir C. G. Heleno, Gustavo O. Morais,\* João L. C. Lopes, Norberto P. Lopes\* and Mauricio G. Constantino\*

Natural products furanoheliangolides can be biomimetically transformed in eremantholides with Stryker's reagent.

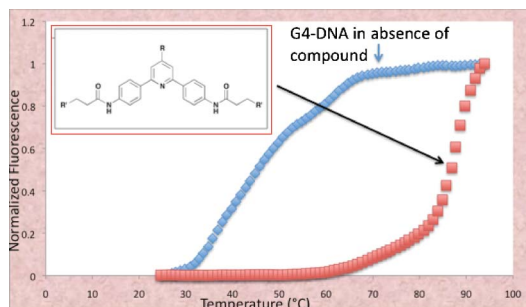


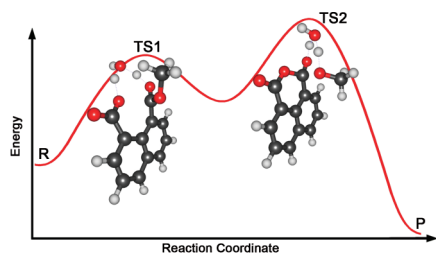
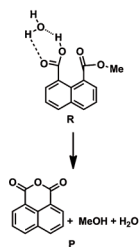
6154

### Unraveling the relationship between structure and stabilization of triarylpyridines as G4-DNA binding ligands

N. M. Smith, Gaëlle Labrunie, Ben Corry, Phong Lan Thao Tran, Marck Norret, Mojgan Djavaheri-Mergny, Colin L. Raston\* and Jean-Louis Mergny\*

Triarylpyridines display a wide range of stabilization for G4-DNA, depending on the nature of the side chains and 4-aryl substituents.





### Mechanism of intramolecular catalysis in the hydrolysis of alkyl monoesters of 1,8-naphthalic acid

Bruno S. Souza, Santiago F. Yunes, Marcelo F. Lima, José C. Gesser, Marcus M. Sá, Haidi D. Fiedler and Faruk Nome\*

Hydrolysis of alkyl 1,8-naphthalic acid monoesters is subject to highly efficient intramolecular nucleophilic catalysis by the neighboring COOH group.

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