

# Organic & Biomolecular Chemistry

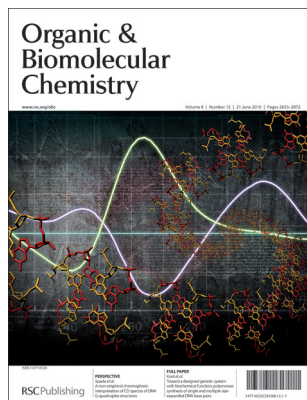
An international journal of synthetic, physical and biomolecular organic chemistry

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## IN THIS ISSUE

ISSN 1477-0520 CODEN OBCRAK 8(12) 2653–2872 (2010)

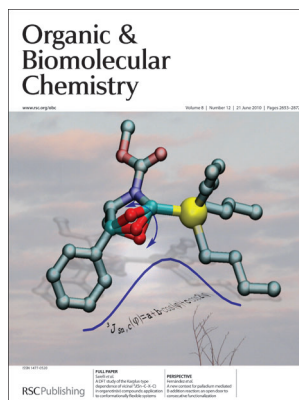


### Cover

See Gian Piero Spada *et al.*, pp. 2683–2692.

Why does the different strand folding in G4-DNAs give rise to different circular dichroism spectra? In this perspective article, the most relevant CD features are rationalised in terms of different stacking orientation between adjacent G-quartets.

Image reproduced by permission of Gian Piero Spada from *Org. Biomol. Chem.*, 2010, **8**, 2683.



### Inside cover

See Giacomo Saielli *et al.*, pp. 2711–2718.

Relativistic DFT calculations, including population analysis, highlight several unexpected features of the Karplus-type dependence of vicinal (Sn–C–X–C) spin-spin couplings in organotin(IV) synthetic intermediates.

Image reproduced by permission of Giacomo Saielli from *Org. Biomol. Chem.*, 2010, **8**, 2711.

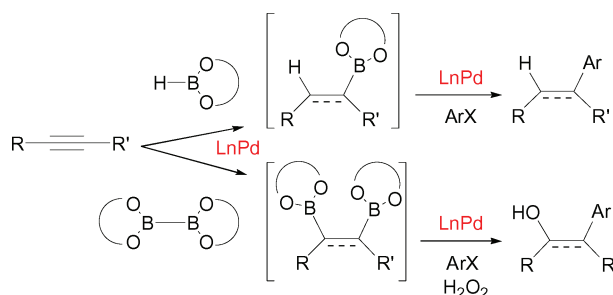
## PERSPECTIVES

2667

### A new context for palladium mediated B-addition reaction: an open door to consecutive functionalization

Cristina Pubill-Ulldemolins, Amadeu Bonet, Carles Bo, Henrik Gulyás\* and Elena Fernández\*

Highlighting the great interest in the application of organoboranes in C–C bond formation, this perspective provides the reader with the criteria to design consecutive tandem organoboron formation/C–C coupling reactions by means of multifaceted palladium catalytic systems.

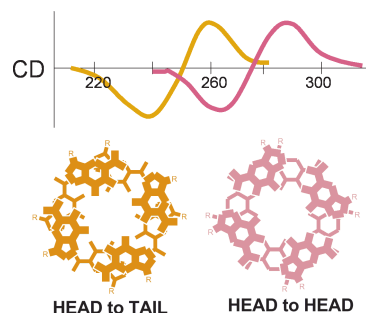


2683

### A non-empirical chromophoric interpretation of CD spectra of DNA G-quadruplex structures

Stefano Masiero, Roberta Trotta, Silvia Pieraccini, Stefano De Tito, Rosaria Perone, Antonio Randazzo\* and Gian Piero Spada\*

While the main difference in CD spectra of G4-DNAs is usually empirically associated to the relative orientation of the strands, in this perspective article, it is rationalised in terms of different stacking orientation (head-to-tail, head-to-head, tail-to-tail) between adjacent G-quartets.



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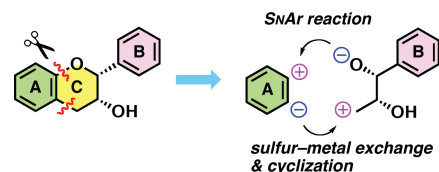
## COMMUNICATIONS

2693

### General synthesis of epi-series catechins and their 3-gallates: reverse polarity strategy

Ken Ohmori, Takahisa Yano and Keisuke Suzuki\*

A general synthetic route to the epi-series catechins was developed based on the reverse polarity strategy. Aromatic nucleophilic substitution reaction followed by the sulfinyl–metal exchange and cyclization enabled stereo-controlled access to various members of epi-series catechins and their 3-gallates.

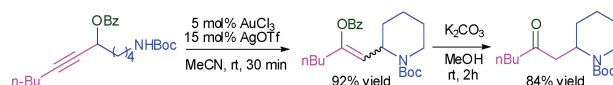


2697

### Gold-catalyzed synthesis of nitrogen-containing heterocycles from $\epsilon$ -*N*-protected propargylic esters

Jianfeng Huang, Xuan Huang and Bo Liu\*

A mild and efficient gold-catalyzed tandem cyclization to piperidiny enol esters has been developed with readily available  $\epsilon$ -*N*-Boc-protected propargylic esters.

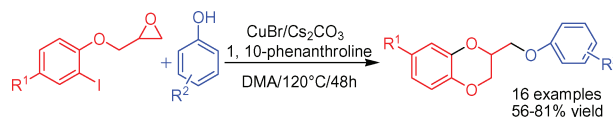


2700

### Copper-catalyzed tandem process: an efficient approach to 2-substituted-1,4-benzodioxanes

Yunyun Liu and Weiliang Bao\*

An efficient method for the preparation of various 2-substituted-1,4-benzodioxanes by CuBr-catalyzed tandem reactions of 2-((*o*-iodophenoxy)methyl)oxiranes with phenols has been developed. The reaction involves the ring-opening process of 2-((2-iodophenoxy)methyl)oxirane followed by an intramolecular C–O cross coupling cyclization.



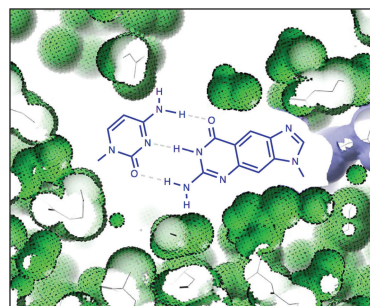
## PAPERS

2704

### Toward a designed genetic system with biochemical function: polymerase synthesis of single and multiple size-expanded DNA base pairs

Haige Lu, Andrew T. Krueger, Jianmin Gao, Haibo Liu and Eric T. Kool\*

Size-expanded DNA (xDNA) represents a new, larger-than-native architecture for a genetic set. The first full study of polymerase-mediated synthesis, editing, and extension of up to four xDNA base pairs is described here. Results show that a repair polymerase (Dpo4) is able to extend xDNA pairs more efficiently than the Klenow polymerase.





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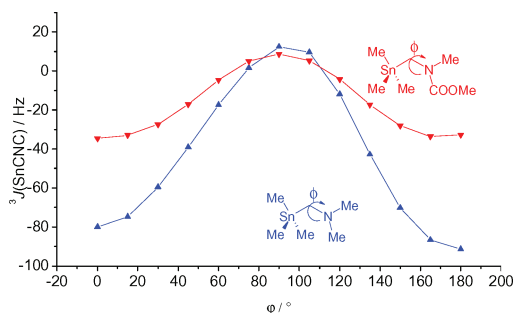
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2711

**A DFT study of the Karplus-type dependence of vicinal  $^3J(\text{Sn}-\text{C}-\text{X}-\text{C})$ ,  $\text{X}=\text{N},\text{O},\text{S}$ , in organotin(IV) compounds: application to conformationally flexible systems**

Girolamo Casella, Francesco Ferrante and Giacomo Saielli\*

Karplus-type curves for Sn–C–X–C couplings have been investigated by relativistic and non-relativistic DFT protocols. The results obtained for simple model systems reveal interesting features which help to rationalize experimental data for cyclic and conformationally flexible  $\alpha$ -aminoorganostannanes.

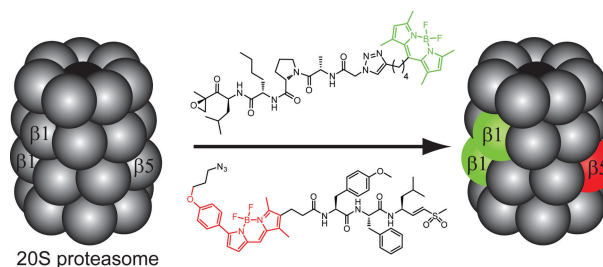


2719

**A panel of subunit-selective activity-based proteasome probes**

Martijn Verdoes, Lianne I. Willems, Wouter A. van der Linden, Boudewijn A. Duivenvoorden, Gijsbert A. van der Marel, Bogdan I. Florea, Alexei F. Kisselev\* and Herman S. Overkleeft\*

The development of fluorescent activity-based probes that selectively target the  $\beta 1$  and  $\beta 5$  active sites of the constitutive proteasome is presented.

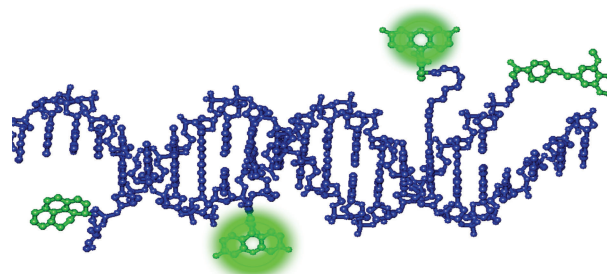


2728

**End-capped HyBeacon probes for the analysis of human genetic polymorphisms related to warfarin metabolism**

Nouha Ben Gaied, James A. Richardson, Daniel G. Singleton, Zhengyun Zhao, David French and Tom Brown\*

End-capped HyBeacon probes have been used to characterize SNPs in genes associated with variations in the efficiency of warfarin metabolism. 5'-Trimethoxystilbene and 3'-pyrene caps increase the differences between hybridised and dissociated states and provide a robust method for interrogation of polymorphic DNA sequences.

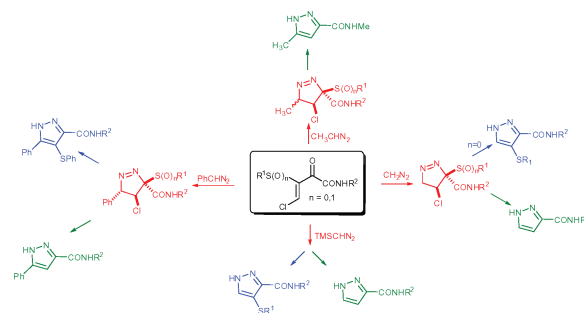


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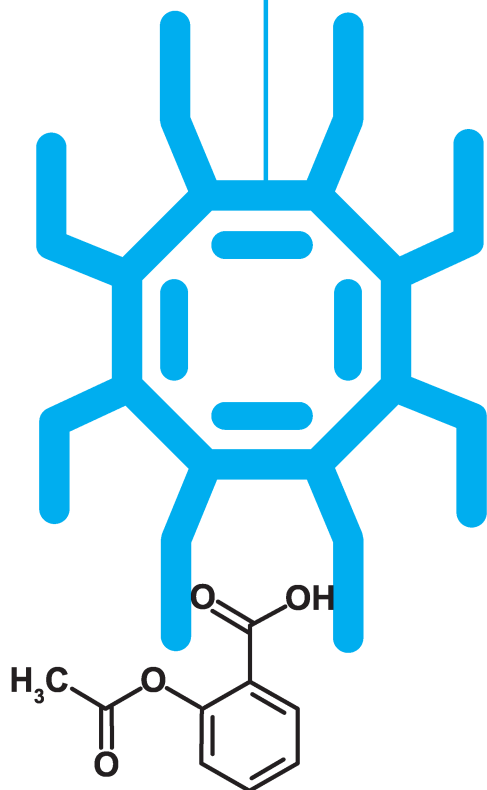
**1,3-Dipolar cycloadditions of 2-thio-3-chloroacrylamides with diazoalkanes**

Marie Kissane, Simon E. Lawrence and Anita R. Maguire\*

2-Thio-3-chloroacrylamides undergo 1,3-dipolar cycloadditions with diazoalkanes leading to a series of novel pyrazolines and pyrazoles. The mechanistic and synthetic features of the cycloadditions to the 2-thio-3-chloroacrylamides at both the sulfide and sulfoxide levels of oxidation are rationalised on the basis of the nature of the substituents.



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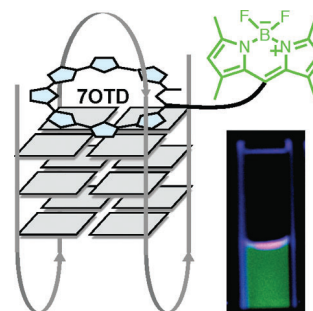
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2749

### Visualization of G-quadruplexes by using a BODIPY-labeled macrocyclic heptaoxazole

Masayuki Tera, Keisuke Iida, Kazunori Ikebukuro, Hiroyuki Seimiya, Kazuo Shin-ya and Kazuo Nagasawa\*

Visualization of G-quadruplex structures could be achieved by fluorescent labeled macrocyclic heptaoxazole namely LIBOD-7OTD in the cell-free and cell-based assay.

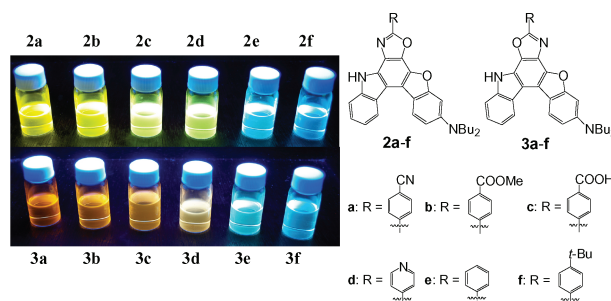


2756

### Synthesis and fluorescence and electrochemical properties of D- $\pi$ -A structural isomers of benzofuro[2,3-*c*]-oxazolo[4,5-*a*]carbazole-type and benzofuro[2,3-*c*]-oxazolo[5,4-*a*]carbazole-type fluorescent dyes

Yosuke Ooyama, Genta Ito, Kohei Kushimoto, Kenji Komaguchi, Ichiro Imae and Yutaka Harima\*

Two heteropolycyclic D- $\pi$ -A structural isomers, which differ in the position of oxygen and nitrogen atoms of the oxazole ring, have been synthesized, and their photophysical and electrochemical properties have been investigated.

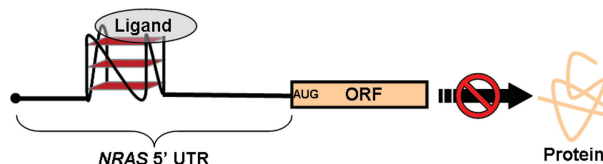


2771

### Small molecule-mediated inhibition of translation by targeting a native RNA G-quadruplex

Anthony Bugaut, Raphaël Rodriguez, Sunita Kumari, Shang-Te Danny Hsu and Shankar Balasubramanian\*

We report on the first example of translation inhibition by a small molecule that targets an RNA G-quadruplex within the 5' UTR of the human *NRAS* proto-oncogene.

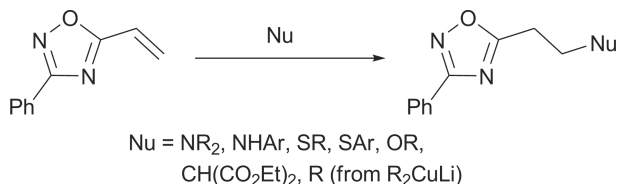


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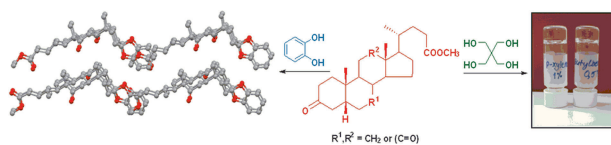
### Tuned methods for conjugate addition to a vinyl oxadiazole; synthesis of pharmaceutically important motifs

Alan R. Burns, Jennifer H. Kerr, William J. Kerr,\* Joanna Passmore, Laura C. Paterson and Allan J. B. Watson

The addition of various nucleophiles to a vinyl 1,2,4-oxadiazole is described. Following optimisation, individual protocols tuned for the use of each specific class of reagent have been developed to allow the installation of nitrogen, sulfur, oxygen, and carbon nucleophiles, and leading to the preparation of a series of compounds containing the pharmaceutically important oxadiazole motif.



2784

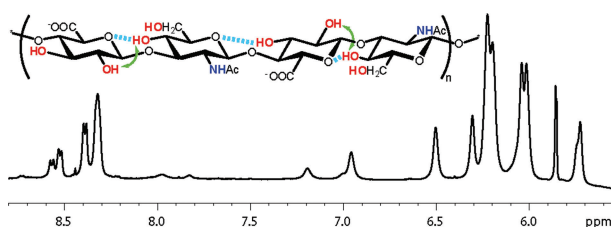


### Bile acid-derived mono- and diketals—synthesis, structural characterization and self-assembling properties

Satu Ikonen,\* Satu Nonappa, Arto Valkonen,  
Raija Juvonen, Hannu Salo and Erkki Kolehmainen

Novel bile acid-derived monoketals of catechol and 2,3-naphthalenediol, as well as mono- and diketals with pentaerythritol have been prepared and characterized. Their self-assembling properties in solution and in the solid state are discussed.

2795

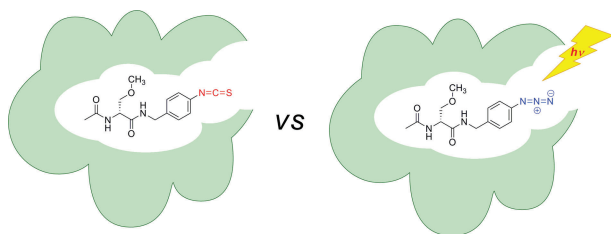


### Experimental evidence of chemical exchange over the $\beta(1 \rightarrow 3)$ glycosidic linkage and hydrogen bonding involving hydroxy protons in hyaluronan oligosaccharides by NMR spectroscopy

Gustav Nestor, Lennart Kenne and Corine Sandström\*

NMR analysis of hydroxy protons of hyaluronan oligosaccharides gives experimental evidence of weak inter-residual hydrogen bond interactions and reveals a chemical exchange interaction between two hydroxy protons over the  $\beta(1 \rightarrow 3)$  glycosidic linkage.

2803

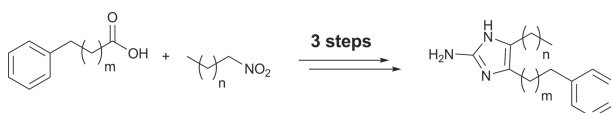


### Proteomic searches comparing two (*R*)-lacosamide affinity baits: An electrophilic arylisothiocyanate and a photoactivated arylazide group

Ki Duk Park, James P. Stables, Rihe Liu\* and Harold Kohn\*

Lacosamide affinity baits were evaluated for their preferential adduction of binding proteins, for the stereospecificity of protein modification, and their use in competition experiments.

2814



### A nitroenolate approach to the synthesis of 4,5-disubstituted-2-aminoimidazoles. Pilot library assembly and screening for antibiotic and antibiofilm activity

Zhaoming Su, Steven A. Rogers, W. Steve McCall,  
Alicia C. Smith, Sindhu Ravishankar, Trey Mullikin and  
Christian Melander\*

4,5-Disubstituted 2-aminoimidazoles with potent antibiotic and antibiofilm activity against terrestrial and marine bacteria.



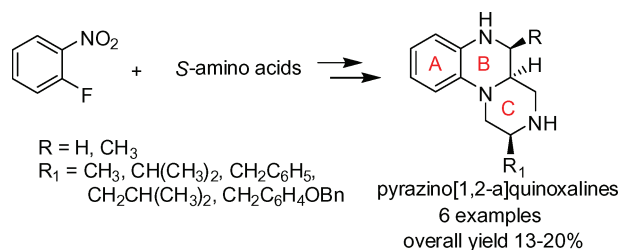
## PAPERS

2823

**A new synthesis of amino acid-based enantiomerically pure substituted 2,3,4,4a,5,6-hexahydro-1*H*-pyrazino[1,2-*a*]quinoxalines**

Krishnananda Samanta and Gautam Panda\*

A new series of enantiomerically pure pyrazino[1,2-*a*]quinoxalines were synthesized for the first time in twelve steps with 13–20% overall yields. Inter and intramolecular Mitsunobu cyclization followed by  $\text{PPh}_3/\text{I}_2$ /imidazole mediated 6-*exo-tet* cyclization were the key steps.

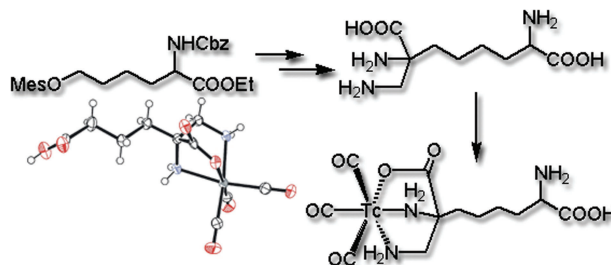


2829

**Syntheses of bifunctional 2,3-diamino propionic acid-based chelators as small and strong tripod ligands for the labelling of biomolecules with <sup>99m</sup>Tc**

Yu Liu, Bruno L. Oliveira, João D. G. Correia, Isabel C. Santos, Isabel Santos, Bernhard Spingler and Roger Alberto\*

2,3-Diamino propionic acid is a strong, albeit scarcely used, tripod ligand. The coupling of different functionalities at  $\alpha$ -carbon leads to bifunctional chelators which can be conjugated to biological vectors for application in molecular imaging.

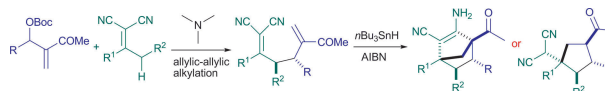


2840

**Tributyltin hydride-mediated radical cyclisation reactions: efficient construction of multiply substituted cyclopentanes**

Jie Lei, Hai-Lei Cui, Rui Li,\* Li Wu, Zheng-Yu Ding and Ying-Chun Chen\*

The  $n\text{Bu}_3\text{SnH}$ -mediated cyclisation reactions of allylic-allylic alkylation products of  $\alpha,\alpha$ -dicyanoalkenes and Morita–Baylis–Hillman (MBH) carbonates of methyl vinyl ketone have been investigated. Cyclopentane derivatives bearing multiple substituents were efficiently prepared with moderate to excellent diastereoselectivity.

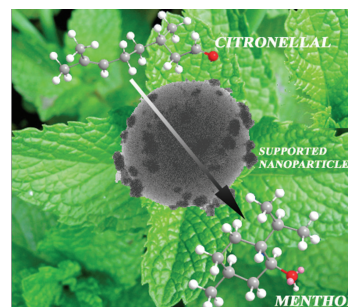


2845

**One-step microwave-assisted asymmetric cyclisation/hydrogenation of citronellal to menthols using supported nanoparticles on mesoporous materials**

Alina Mariana Balu, Juan Manuel Campelo, Rafael Luque\* and Antonio Angel Romero

A microwave-assisted one step cyclisation/hydrogenation methodology was able to provide high yields of menthols from citronellal using a simple and environmentally friendly heterogeneous catalyst based on supported nanoparticles on porous supports.



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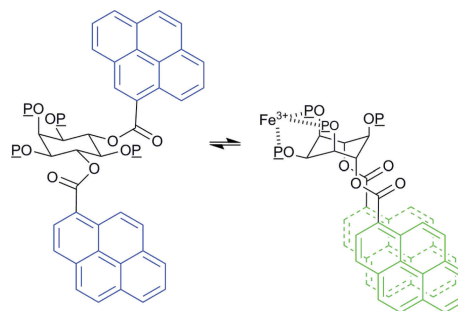
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2850

### Conformational analysis of the natural iron chelator *myo*-inositol 1,2,3-trisphosphate using a pyrene-based fluorescent mimic

D. Mansell, N. Rattray, L. L. Etchells, C. H. Schwalbe, A. J. Blake, J. Torres, C. Kremer, E. V. Bichenkova, C. J. Barker and S. Freeman\*

The selective interaction of  $\text{Fe}^{3+}$  with a *myo*-inositol phosphate containing the natural antioxidant 1,2,3-trisphosphate motif has been monitored using a pyrene-based fluorescent probe: the penta-equatorial chair (blue fluorescence) ring flips to the unstable penta-axial conformation (green excimer fluorescence) upon binding  $\text{Fe}^{3+}$ .

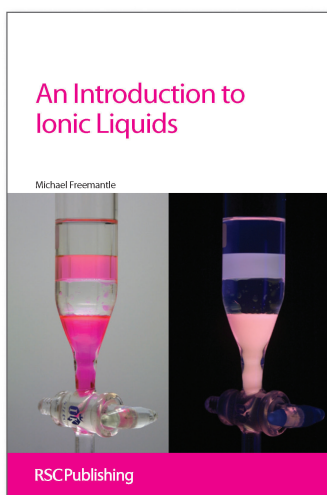
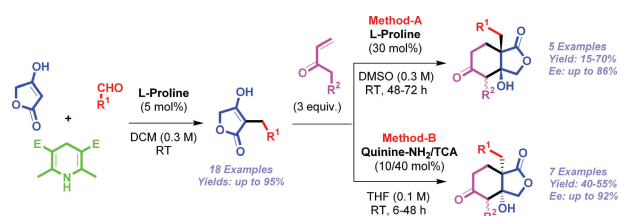


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### Direct catalytic asymmetric synthesis of highly functionalized tetronic acids/tetrahydro-isobenzofuran-1,5-diones *via* combination of cascade three-component reductive alkylations and Michael-aldol reactions

Dhevalapally B. Ramachary\* and Mamillapalli Kishor

A general process for the synthesis of tetrahydro-isobenzofuran-1,5-diones was achieved for the first time through asymmetric cascade Michael-aldol reaction of 4-hydroxy-3-alkyl-5*H*-furan-2-ones with alkyl vinyl ketones in the presence of a catalytic amount of L-proline or Q-NH<sub>2</sub>/TCA



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Michael Freemantle

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