

# Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry  
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## IN THIS ISSUE

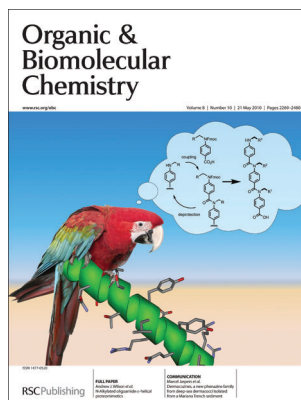
ISSN 1477-0520 CODEN OBCRAK 8(10) 2269–2480 (2010)



### Cover

See Kay Severin *et al.*, pp. 2327–2331.  
 A dynamic mixture of iron complexes can be used as a colorimetric sensor for sulfated glycosaminoglycans such as heparin.

Image reproduced by permission of Kay Severin from *Org. Biomol. Chem.*, 2010, **10**, 2327.



### Inside cover

See Andrew J. Wilson *et al.*, pp. 2344–2351.  
 'Who's a pretty helix?' On page 2344 of this issue, Wilson and co-workers describe the solid-phase synthesis of a novel series of oligobenzamide  $\alpha$ -helix mimetics shown to act as potent inhibitors of the p53-hDM2 interaction.

Image reproduced by permission of Andrew J. Wilson from *Org. Biomol. Chem.*, 2010, **10**, 2344.

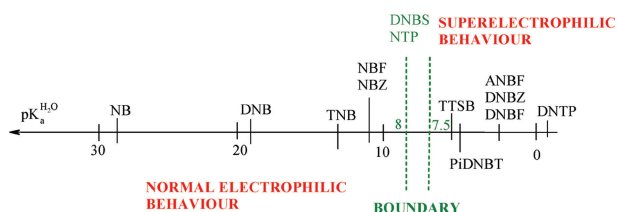
## PERSPECTIVE

2285

### Assessing the superelectrophilic dimension through $\sigma$ -complexation, $S_NAr$ and Diels–Alder reactivity

Erwin Bunzel and François Terrier

Investigation of highly electron-deficient heteroaromatic structures allows extension of the classical domain of reactivity in  $S_NAr$  and  $\sigma$ -complexation processes by 13 orders of magnitude, as illustrated by the  $pK_a^{H_2O}$  scale for covalent hydration. This provides access to a superelectrophilic dimension of major importance in terms of synthetic applications and recognition of new structure–reactivity relationships.



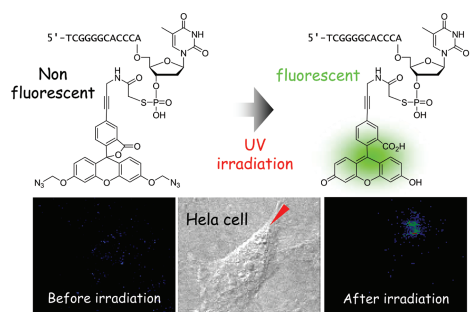
## COMMUNICATIONS

2309

### Photoactivatable fluorescein derivatives with azidomethyl caging groups for tracing oligonucleotides in living human cells

Kazuhiro Furukawa, Hiroshi Abe,\* Satoshi Tsuneda and Yoshihiro Ito\*

A new photocaged fluorescent compound, azidomethyl fluorescein, was successfully utilized to monitor the dynamics of oligonucleotides in living human cells.



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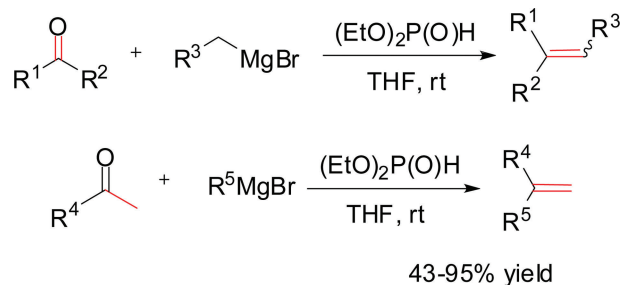
## COMMUNICATIONS

2312

**A novel and efficient method for the olefination of carbonyl compounds with Grignard reagents in the presence of diethyl phosphite**

Tongqiang Wang, Yuanyuan Hu and Songlin Zhang\*

A one-pot manner of carbonyl olefination: a range of conjugated dienes, terminal olefins, multisubstituted-alkenes and conjugated enynes could be readily obtained in good to excellent yields in mild conditions.

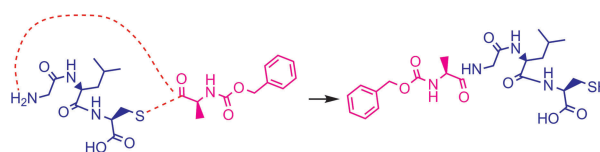


2316

**The chemical ligation of selectively *S*-acylated cysteine peptides to form native peptides via 5-, 11- and 14-membered cyclic transition states**

Alan R. Katritzky,\* Nader E. Abo-Dya, Srinivasa R. Tala and Zakaria K. Abdel-Samii

*N*-Pg-Cysteine peptides are *S*-acylated by *N*-(Pg- $\alpha$ -aminoacyl)benzotriazoles and *N*-Fmoc protected *S*-acyl-isopeptides were deprotected to *S*-acyl-isopeptides. The *S*-acyl-isodi-, isotetra-, and isopenta-peptides undergo ligation via 5-, 11-, and 14-membered transition states to give native peptides.

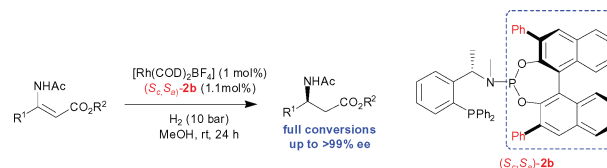


2320

**Chiral 1-phenylethylamine-derived phosphine-phosphoramidite ligands for highly enantioselective Rh-catalyzed hydrogenation of  $\beta$ -(acylamino)acrylates: significant effect of substituents on 3,3'-positions of binaphthyl moiety**

Xiao-Mao Zhou, Jia-Di Huang, Li-Bin Luo, Chen-Lu Zhang, Xiang-Ping Hu\* and Zhuo Zheng

New phosphine-phosphoramidite ligands were successfully applied in the Rh-catalyzed asymmetric hydrogenation of  $\beta$ -(acylamino)acrylates.

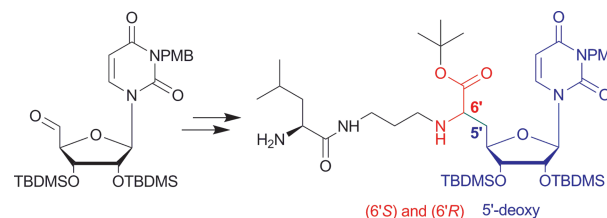


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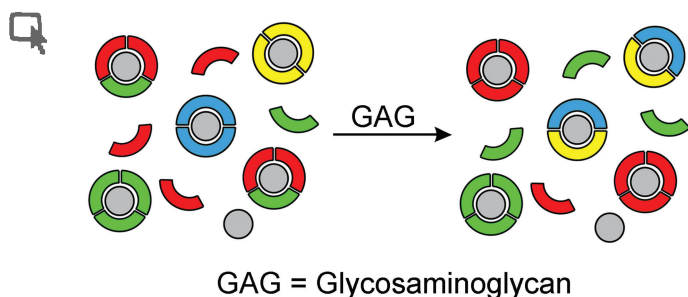
**Novel 5'-deoxy nucleosyl amino acid scaffolds for the synthesis of muraymycin analogues**

Anatol P. Spork and Christian Ducho\*

Naturally occurring nucleoside antibiotics such as muraymycins represent promising lead structures for the development of novel antibacterial agents. A concise synthesis of 5'-deoxy muraymycin derivatives has been developed. The key step was the highly stereoselective asymmetric hydrogenation of suitable dihydro amino acid precursors, providing unique nucleosyl amino acid structures.



2327

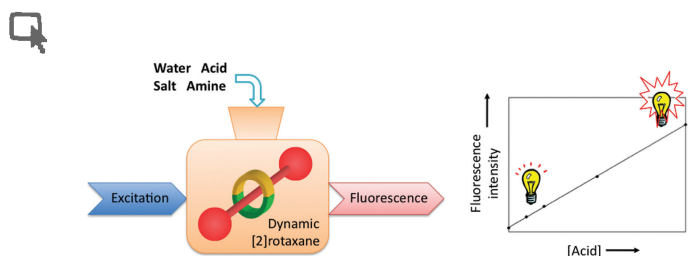


### Pattern-based sensing of sulfated glycosaminoglycans with a dynamic mixture of iron complexes

Peter-Korbinian Müller-Graff, Helga Szelke, Kay Severin\* and Roland Krämer\*

A dynamic mixture of Fe(II) complexes was used as a colorimetric sensor for sulfated glycosaminoglycans such as heparin, dextran sulfate, chondroitin sulfate, and heparan sulfate.

2332

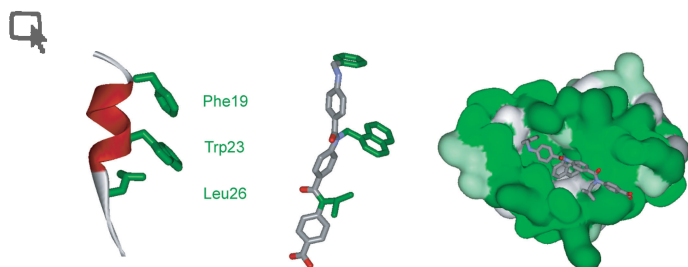


### Self-assembly, stability quantification, controlled molecular switching, and sensing properties of an anthracene-containing dynamic [2]rotaxane

Wing-Yan Wong, Ken Cham-Fai Leung\* and J. Fraser Stoddart\*

A novel anthracene-containing dynamic [2]rotaxane has been synthesised and characterised. The stability of the [2]rotaxane, which has been investigated after the addition of water, acids, salts, and an amine, is monitored by observing the anthracene fluorescence and  $^1\text{H}$  NMR chemical shifts. The [2]rotaxane has been shown to be a good acid sensor.

2344

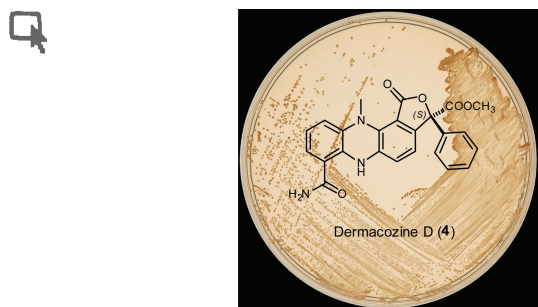


### N-alkylated oligoamide $\alpha$ -helical proteomimetics

Frederick Campbell, Jeffrey P. Plante, Thomas A. Edwards,\* Stuart L. Warriner\* and Andrew J. Wilson\*

In this paper, the design, synthesis and testing of a novel class of proteomimetic is described. Potent  $\mu\text{M}$  inhibitors of the p53-*h*DM2 protein-protein interaction are identified.

2352



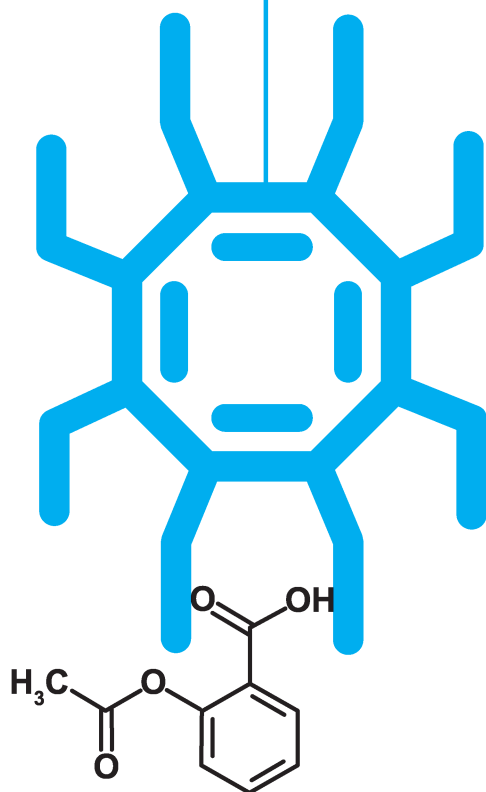
### Dermacozines, a new phenazine family from deep-sea dermacocci isolated from a Mariana Trench sediment

Wael M. Abdel-Mageed, Bruce F. Milne, Marcell Wagner, Marc Schumacher, Peter Sandor, Wasu Pathom-aree, Michael Goodfellow, Alan T. Bull, Koki Horikoshi, Rainer Ebel, Marc Diederich, Hans-Peter Fiedler and Marcel Jaspars\*

Seven phenazines with unique modifications were produced by the piezotolerant actinobacterium *Dermacoccus abyssi* which was isolated from a sediment sample collected from the deepest place on earth, Challenger Deep in the Mariana Trench.



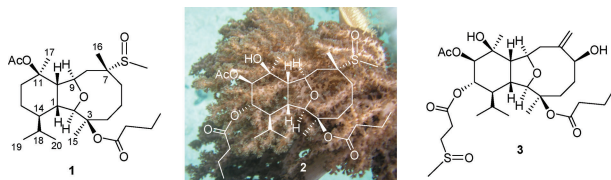
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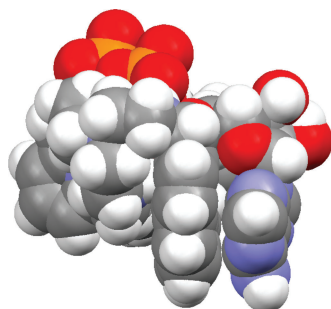


### Anti-inflammatory eunicellin-based diterpenoids from the cultured soft coral *Klyxum simplex*

Bo-Wei Chen, Chih-Hua Chao, Jui-Hsin Su, Zhi-Hong Wen, Ping-Jyun Sung and Jyh-Horng Sheu\*

Three novel eunicellin-based diterpenoids, namely klysimplexin sulfoxides A–C, were isolated from the cultured soft coral *Klyxum simplex*.

2367

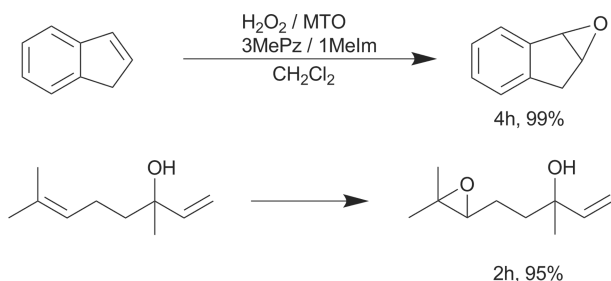


### Tritopic phenanthroline and pyridine tail-tied aza-scorpians

Jorge González, José M. Llinares, Raquel Belda, Javier Pitarch, Concepción Soriano, Roberto Tejero, Begoña Verdejo and Enrique García-España\*

Two new tail-tied aza-macrocycles in which two pyridinophane scorpion equivalent units have been covalently connected through 2,6-dimethylpyridine or 2,9-dimethylphenanthroline linkages show interesting capacity as  $Zn^{2+}$  and polyphosphate anion binders.

2377

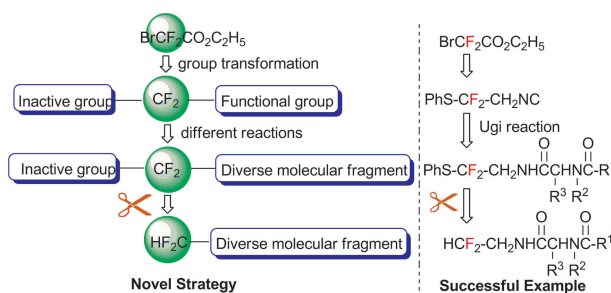


### An effective procedure for the synthesis of acid-sensitive epoxides: Use of 1-methylimidazole as the additive on methyltrioxorhenium-catalyzed epoxidation of alkenes with hydrogen peroxide

Shigekazu Yamazaki\*

The combined use of 3-methylpyrazole and 1-methylimidazole as the additives for methyltrioxorhenium(MTO)-catalyzed epoxidation has been found to be an effective procedure for the synthesis of acid-sensitive epoxides in excellent yields.

2386



### A general strategy for construction of a difluoromethyl compound library and its application in synthesis of pseudopeptides bearing a terminal difluoromethyl group

Jingjing Wu, Song Cao,\* Nianjin Liu, Li Shen, Jinlong Yu, Jian Zhang, Hui Li and Xuhong Qian

A novel synthesis strategy that uses common reaction conditions to transform a collection of simple building blocks into complex molecules bearing a terminal difluoromethyl group was described. The strategy is illustrated by application to the synthesis of  $CF_2H$ -bearing pseudopeptides via Ugi reaction.

## Dalton Discussion 12: Catalytic C-H and C-X Bond Activation

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Organised jointly by the Dalton Division and Organic Division, DD12 will bring together the organic, organometallic and inorganic (coordination chemistry) communities from academia and industry to discuss the current state of the art, the development and future of late metal-catalysed cross-coupling strategies involving C-X and/or C-H bonds.

The meeting will highlight the importance of catalytic bond activation in cross-coupling chemistry. The latest research will be presented and discussed.

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- Synthetic chemistry (including applications)
- Inorganic and organometallic chemistry
- Reaction mechanism (physical organic and organometallic chemistry)
- Transition metal catalysis
- Applications of C-H and C-X bond activation in organic synthesis

### Keynote speakers

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*The University of British Columbia, Canada*

**William D. Jones**  
*University of Rochester, USA*

**Aiwen Lei**  
*Wuhan University, China*

**Zhang-jie Shi**  
*Peking University, China*

### Invited speakers

**Robin Bedford**  
*University of Bristol, UK*

**John M. Brown**  
*University of Oxford, UK*

**Stuart Macgregor**  
*Heriot-Watt University, Edinburgh, UK*

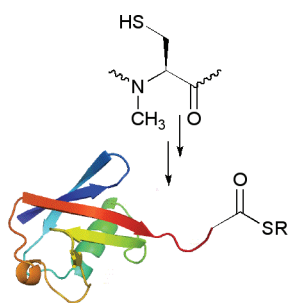
**Hans de Vries**  
*DSM Pharmaceutical Products, The Netherlands*

**Offers of contributed papers related to the listed themes for poster presentation are invited by 16 July 2010.**

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2392

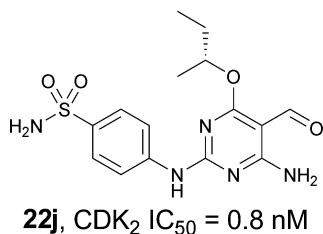


### *N*-Methylcysteine-mediated total chemical synthesis of ubiquitin thioester

Lesly A. Erlich, K. S. Ajish Kumar, Mahmood Haj-Yahya, Philip E. Dawson and Ashraf Brik\*

A new method for the synthesis of ubiquitin thioester was developed. The strategy was applied to the preparation of ubiquitylated  $\alpha$ -synuclein(1–17), which was shown to be a substrate for the ubiquitin C-terminal hydrolase.

2397

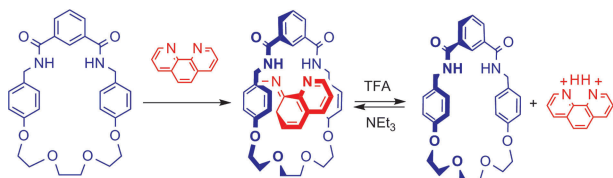


### Synthesis and biological evaluation of 5-substituted *O*<sup>4</sup>-alkylpyrimidines as CDK2 inhibitors

Francesco Marchetti, Céline Cano, Nicola J. Curtin, Bernard T. Golding, Roger J. Griffin, Karen Haggerty, David R. Newell, Rachel J. Parsons, Sara L. Payne, Lan Z. Wang and Ian R. Hardcastle\*

CDK2 inhibitory structure–activity relationships have been explored for a range of 5-substituted *O*<sup>4</sup>-alkylpyrimidines. The 5-formyl derivatives show selectivity for CDK-2 over other CDK family members.

2408

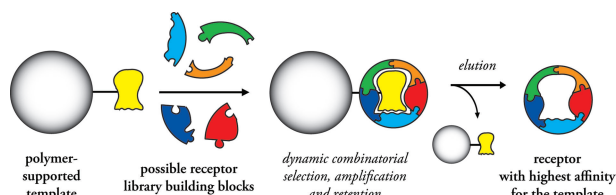


### Acid/base controllable molecular switch based on a neutral phenanthroline guest penetrated pseudorotaxane

Masahiro Muraoka,\* Hiromitsu Irie and Yohji Nakatsuji\*

New pseudorotaxanes incorporating a bisamide macrocycle and neutral phenanthroline derivatives can be formed by hydrogen bonding and  $\pi$ -electron interaction, which gives a pH controllable reversible molecular switching system.

2414



### Affinity chromatography in dynamic combinatorial libraries: one-pot amplification and isolation of a strongly binding receptor

Pol Besenius,\* Peter A. G. Cormack,\* R. Frederick Ludlow, Sijbren Otto and David C. Sherrington

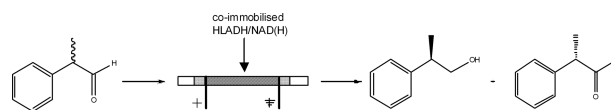
We report the one-pot amplification and isolation of a nanomolar receptor in a multibuilding block aqueous dynamic combinatorial library consisting of well over 140 theoretical members using a polymer-bound template.

2419

### The development and evaluation of a conducting matrix for the electrochemical regeneration of the immobilised co-factor NAD(H) under continuous flow

B. Ngamsom, A. M. Hickey, G. M. Greenway,\*  
J. A. Littlechild, T. McCreedy, P. Watts and C. Wiles

Employing a novel conducting controlled pore glass-poly(pyrrole) material for the co-immobilisation of HLADH and NAD(H), we were able to fabricate a reagent-less flow reactor capable of the continuous biosynthesis of chiral compounds under an applied voltage.

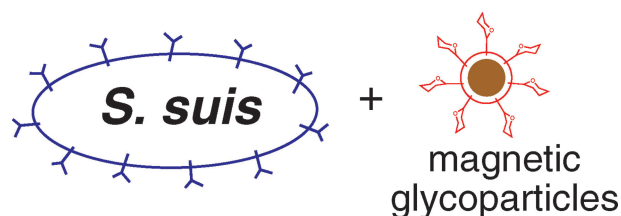


2425

### Detection of pathogenic *Streptococcus suis* bacteria using magnetic glycoparticles

Núria Parera Pera, Annika Kouki, Sauli Haataja,  
Hilbert M. Branderhorst, Rob M. J. Liskamp,  
Gerben M. Visser, Jukka Finne and Roland J. Pieters\*

Magnetic glycoparticles can be used to bind, concentrate, and detect the pathogenic bacterium *Streptococcus suis*

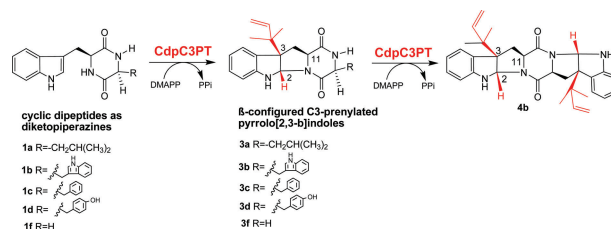


2430

### Preparation of pyrrolo[2,3-b]indoles carrying a $\beta$ -configured reverse C3-dimethylallyl moiety by using a recombinant prenyltransferase CdpC3PT

Wen-Bing Yin, Xia Yu, Xiu-Lan Xie and Shu-Ming Li\*

Six  $\beta$ -configured reversely C3-prenylated pyrrolo[2,3-b]indoles were successfully prepared from cyclic tryptophan-containing dipeptides by using a recombinant prenyltransferase from *Neosartorya fischeri*.

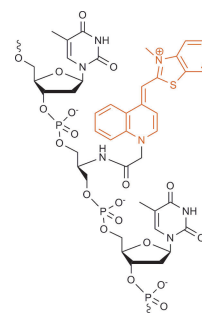


2439

### Designed thiazole orange nucleotides for the synthesis of single labelled oligonucleotides that fluoresce upon matched hybridization

Lucas Bethge, Ishwar Singh and Oliver Seitz\*

DNA conjugates that contain thiazole orange as an artificial base were synthesized and studied by fluorescence spectroscopy, revealing that fluorescence-on-hybridization can only be obtained through careful design of the backbone structure.





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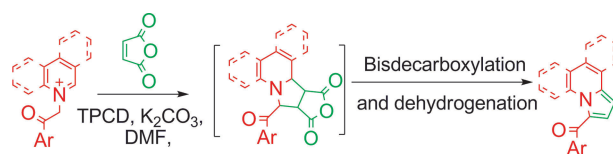
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2449

### Regioselective synthesis of 3-acylindolizines and benzo-analogues *via* 1,3-dipolar cycloadditions of *N*-ylides with maleic anhydride

Yun Liu, Yan Zhang, Yong-Miao Shen, Hong-Wen Hu and Jian-Hua Xu\*

3-Acylindolizines and their benzo- analogues are regioselectively synthesized by one pot reactions of the *N*-ylide with maleic anhydride, taking advantage of the oxidative bisdecarboxylation and dehydrogenation of the primary cycloadducts by the mild oxidant tetrakispyridinecobalt(II) dichromate (TPCD).

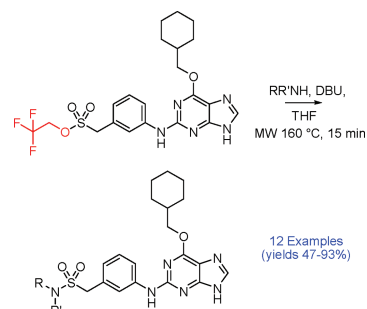


2457

### Synthesis of sulfonamide-based kinase inhibitors from sulfonates by exploiting the abrogated $S_N2$ reactivity of 2,2,2-trifluoroethoxysulfonates

Christopher Wong, Roger J. Griffin, Ian R. Hardcastle, Julian S. Northen, Lan-Zhen Wang and Bernard T. Golding\*

The reduced  $S_N2$  reactivity of the 2,2,2-trifluoroethyl group has been used for the synthesis of 6-cyclohexylmethoxy-2-aryl-aminopurines with a sulfonamide moiety attached to the aryl ring *via*  $CH_2$ ; such compounds are inhibitors of kinases relevant to cancer treatment.

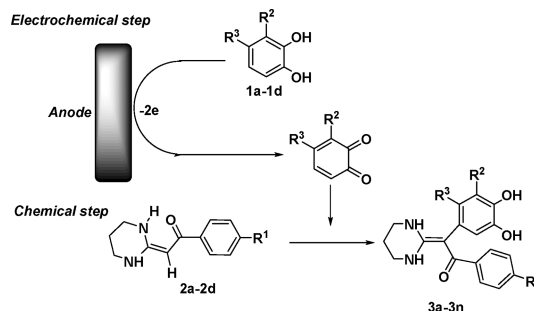


2465

### Anodic oxidation of catechols in the presence of $\alpha$ -oxoketene *N,N*-acetals with a tetrahydropyrimidine ring: selective $\alpha$ -arylation reaction

Cheng-Chu Zeng,\* Da-Wei Ping, Li-Ming Hu, Xiu-Qing Song and Ru-Gang Zhong

This paper provides an efficient way to obtain  $\alpha$ -aryl  $\alpha$ -oxoketene *N,N*-acetals with a tetrahydropyrimidine ring.

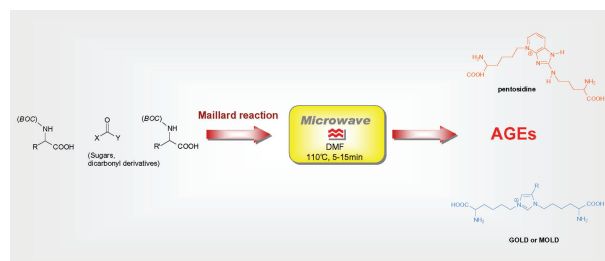


2473

### Microwave-assisted Maillard reactions for the preparation of advanced glycation end products (AGEs)

Sonja Visentin, Claudio Medana, Alessandro Barge, Valeria Giancotti and Giancarlo Cravotto\*

A MW-assisted Maillard reaction in DMF showed to be a straightforward method for the preparation of pentosidine and other AGE derivatives paving the road to a plethora of challenging scientific applications.



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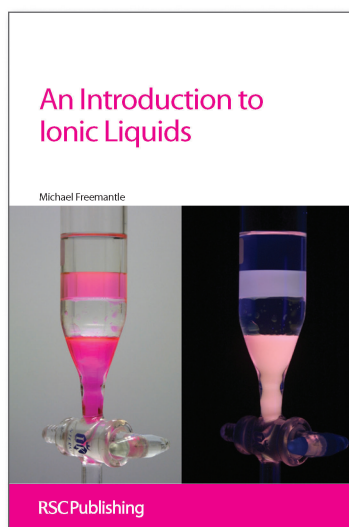
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