

# Organic & Biomolecular Chemistry

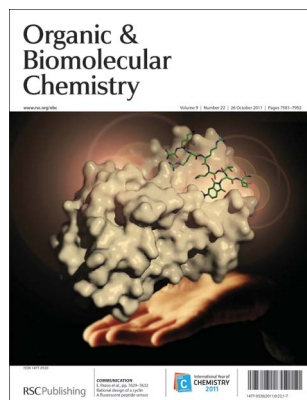
An international journal of synthetic, physical and biomolecular organic chemistry

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## IN THIS ISSUE

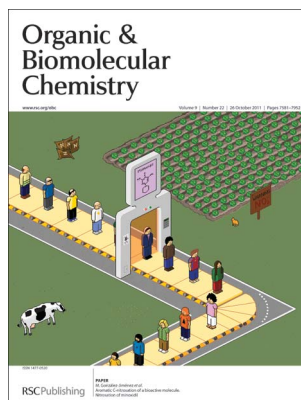
ISSN 1477-0520 CODEN OBCRAK 9(22) 7581–7952 (2011)



### Cover

See E. Pazos *et al.*, pp. 7629–7632.

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### Inside cover

See M. González-Jiménez *et al.*, pp. 7680–7684.

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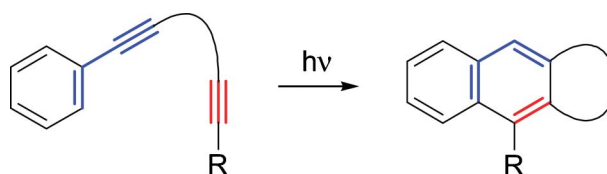
## EMERGING AREA

7599

### The photo-dehydro-Diels–Alder (PDDA) reaction

Pablo Wessig,\* Annika Matthes and Charlotte Pick

The synthetic scope of the photo-dehydro-Diels–Alder reaction for the construction of aromatic rings, especially naphthalenes, is demonstrated on the basis of various examples.



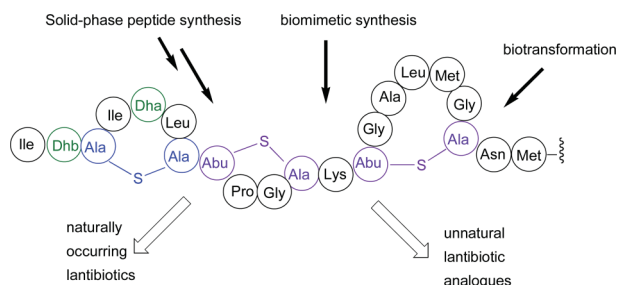
## PERSPECTIVE

7606

### The challenge of the lantibiotics: synthetic approaches to thioether-bridged peptides

Alethea B. Tabor\*

The unique antibacterial properties and structural complexity of the lantibiotics has stimulated considerable interest in developing methodology to synthesise these peptides, and several successful approaches have recently been reported.



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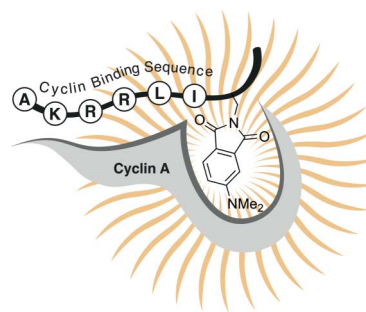
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7629

**Rational design of a cyclin A fluorescent peptide sensor**

Elena Pazos, Miguel Pérez, Hugo Gutiérrez-de-Terán, Mar Orzáez, Tatiana Guevara, José L. Mascareñas\* and M. Eugenio Vázquez\*

We report the development of a fluorescent sensor designed to target cyclin A, which plays a key role in the regulation of the cell cycle. Computational studies explain the observed emission increase, suggesting that the 4-DMAP fluorophore in the peptide is protected from the bulk solvent when inserted into the hydrophobic binding groove of cyclin A.

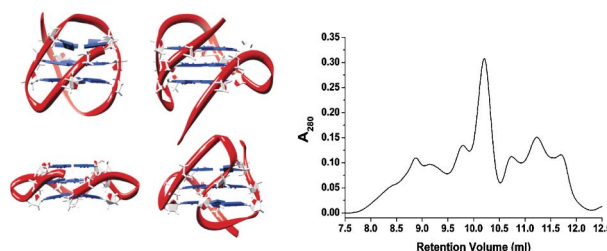


7633

**Polymorphism and resolution of oncogene promoter quadruplex-forming sequences**

M. Clarke Miller, Huy T. Le, William L. Dean, Patrick A. Holt, Jonathan B. Chaires and John O. Trent\*

We report the separation of several quadruplex species formed by ten oncogene promoter sequences by Size Exclusion Chromatography (SEC). SEC enables an unperturbed view of the structural polymorphism inherent to quadruplex formation.

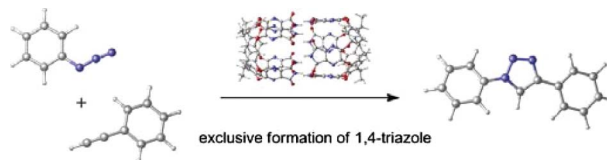


7638

**On the enhanced reactivity and selectivity of triazole formation in molecular flasks. A theoretical rationale**

David Cantillo,\* Martín Ávalos, Reyes Babiano, Pedro Cintas, José L. Jiménez and Juan C. Palacios

An ONIOM computational methodology satisfactorily rationalizes the experimental features of an azide-alkyne cycloaddition conducted within a supramolecular flask.

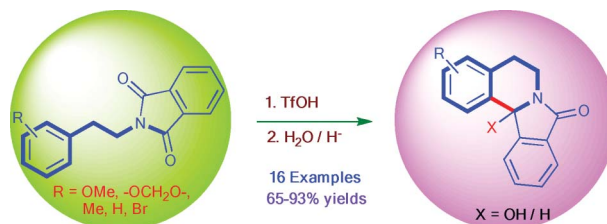


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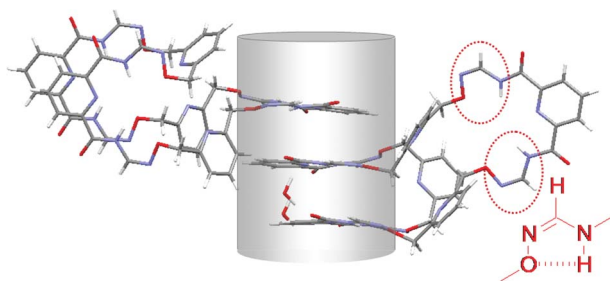
**Brønsted acid assisted activation of imide carbonyl group: regioselective synthesis of isoindoloisoquinolinone alkaloid (±)-nuevamine**

Jayaraman Selvakumar and Chinnasamy Ramaraj Ramanathan\*

Super acid, trifluoromethanesulfonic acid, activation of imide carbonyl group facilitates the construction of isoindoloisoquinolinone derivatives and regioselective synthesis of the alkaloid (±)-nuevamine.



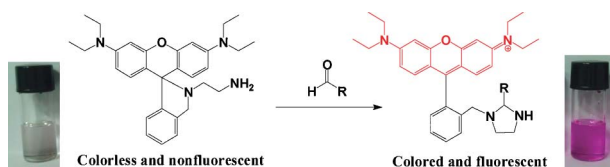
7647

**Z-Formamidoximes in molecular folding and macrocycles**

Weiwen Zhao, Ruiyao Wang and Anne Petitjean\*

The formamidoxime Z isomer coupled with the pyridylbiscarboxamide conformational codon fold into planar, curved units prone to self-assembly into channel-like structures, as exemplified with original macrocycles.

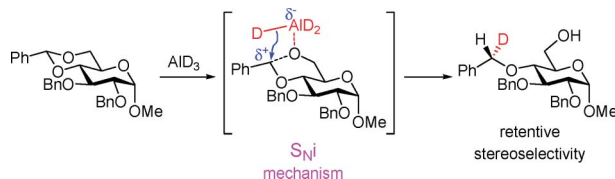
7652

**Chromo-fluorogenic detection of aldehydes with a rhodamine based sensor featuring an intramolecular deoxylactam**

Zhu Li, Zhongwei Xue, Zhisheng Wu, Jiahuai Han and Shoufa Han\*

A chromo-fluorogenic detection of aldehydes was achieved by a rhodamine-deoxylactam based sensor, allowing chemoselective labeling of cell surface sialoproteins.

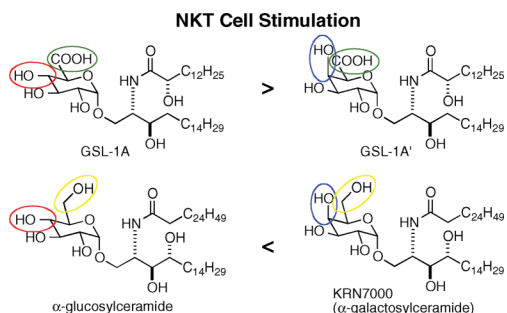
7655

**Deuterium-isotope study on the reductive ring opening of benzylidene acetals**

I-Chi Lee, Medel Manuel L. Zulueta, Chi-Rung Shie, Susan D. Arco and Shang-Cheng Hung\*

Deuterated reference compounds were prepared and subsequently utilized in probing the stereoselectivity of the reductive ring opening of benzylidene acetals.

7659

**Impact of sugar stereochemistry on natural killer T cell stimulation by bacterial glycolipids**

Shenglou Deng, Jochen Mattner, Zhuo Zang, Li Bai, Luc Teyton, Albert Bendelac and Paul B. Savage\*

Carboxylate groups at C6'' in glycolipids alter the *galacto* vs. *gluco* stereoselectivity of recognition and cytokine release by natural killer T cells as compared to glycolipids containing unoxidized sugars.



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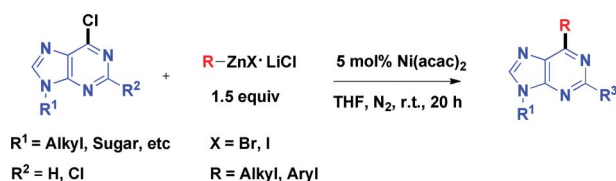
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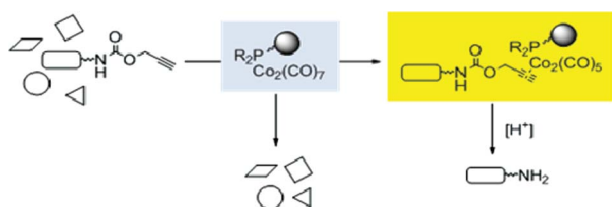


### Nickel-catalyzed Negishi cross-couplings of 6-chloropurines with organozinc halides at room temperature

Dong-Chao Wang, Hong-Ying Niu, Gui-Rong Qu,\*  
Lei Liang, Xue-Jiao Wei, Yang Zhang and Hai-Ming Guo\*

An efficient method for the synthesis of 6-alkyl or 6-aryl purines was developed *via* ligand-free nickel-catalyzed Negishi cross-couplings of 6-chloropurines and organozinc halides, giving good to excellent yields.

7667

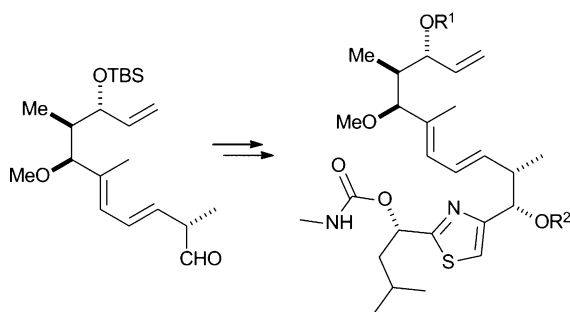


### Catch and release of alkyne-tagged molecules in water by a polymer-supported cobalt complex

Hiromichi Egami, Shinji Kamisuki, Kosuke Dodo,  
Miwako Asanuma, Yoshitaka Hamashima and  
Mikiko Sodeoka\*

Direct catch and release reactions of an alkyne-tagged molecule in aqueous media were demonstrated using the PS-PEG-supported cobalt carbonyl complex, under mild conditions compatible to biochemical experiments.

7671

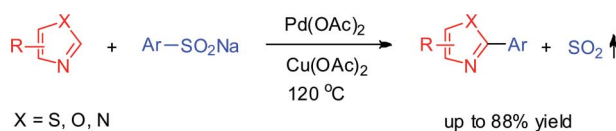


### Synthesis and activity of the archazolid western hemisphere

Ann B. Tran, Geoffrey C. Melly, Ryan Doucette,  
Brook Ashcraft, Leanne J. Sebren, Nathan Havko,  
Jeffery C. Young and Gregory W. O'Neil\*

A synthesis of the archazolid western hemisphere was completed and its V-ATPase inhibitory activity evaluated using a convenient plant-based assay.

7675



### Palladium-catalyzed desulfitative C–H arylation of azoles with sodium sulfonates

Ru Chen, Saiwen Liu, Xinhua Liu, Luo Yang and  
Guo-Jun Deng\*

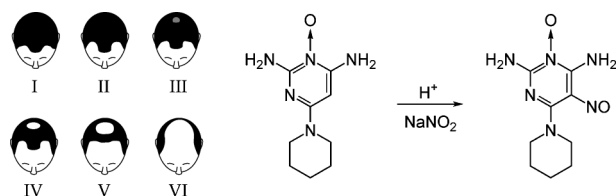
A palladium-catalyzed desulfitative C–H arylation of azoles with sodium sulfonates using  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  as oxidant has been discovered. The reaction proceeded well for a range of different substrates under oxidative conditions.

7680

### Aromatic C-nitrosation of a bioactive molecule. Nitrosation of minoxidil

Mario González-Jiménez, Jorge Arenas-Valgañón, Emilio Calle and Julio Casado\*

Minoxidil is a bioactive molecule widely used as an antihypertensive and antialopecia agent. The *N*-nitrosominoxidil resulting from its nitrosation could be potentially harmful to the minoxidil users.

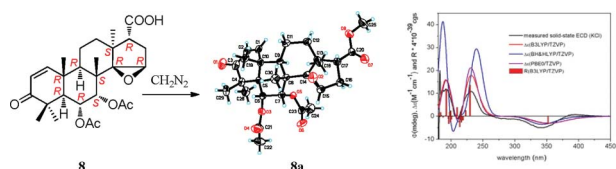


7685

### Protolimonoids and norlimonoids from the stem bark of *Toona ciliata* var. *pubescens*

Jian-Rong Wang, Hai-Li Liu, Tibor Kurtán, Attila Mándi, Sándor Antus, Jia Li, Hai-Yan Zhang and Yue-Wei Guo\*

Nine new protolimonoids and norlimonoids, three of them possessing unprecedented skeletons, were isolated and characterized from *Toona ciliata* var. *pubescens*.

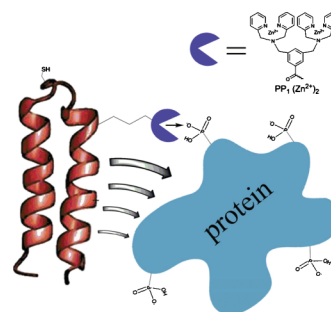


7697

### The molecular recognition of phosphorylated proteins by designed polypeptides conjugated to a small molecule that binds phosphate

Adam T. Ślósarczyk and Lars Baltzer\*

The conjugation of designed polypeptides to a small organic molecule as a strategy for the recognition and binding of phosphorylated proteins.

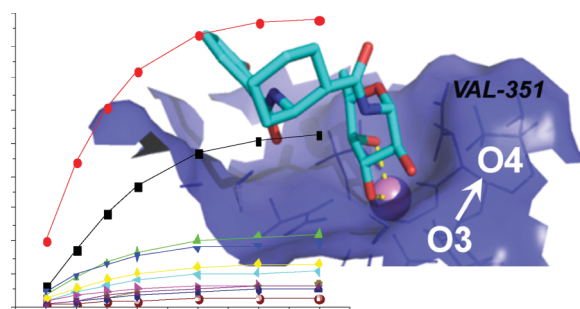


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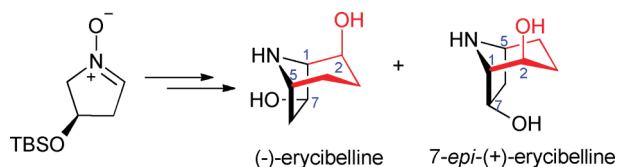
### Insights into molecular recognition of Lewis<sup>X</sup> mimics by DC-SIGN using NMR and molecular modelling

Cinzia Guzzi, Jesús Angulo, Fabio Doro, José J. Reina, Michel Thépaut, Franck Fieschi, Anna Bernardi, Javier Rojo and Pedro M. Nieto\*

STD and modelling studies have been carried out to define the complexes of fucosylamides and DC-SIGN ECD revealing additional interactions with the aromatic moiety of the ligand.



7713

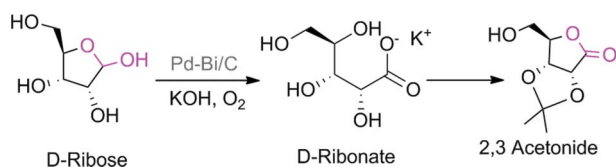


### A concise stereoselective synthesis of (-)-erycibelline

Zhao-Lan Zhang, Shinpei Nakagawa, Atsushi Kato, Yue-Mei Jia, Xiang-Guo Hu and Chu-Yi Yu\*

(-)-Erycibelline, the dihydroxynortropane alkaloid isolated from *Erycibe elliptimba* Merr. et Chun., was synthesized using a cyclic nitronium as advanced intermediate, wherein the key step was the  $\text{SmI}_2$ -induced intramolecular reductive coupling of cyclic nitronium with aldehyde which resulted in good yield and stereoselectivity.

7720

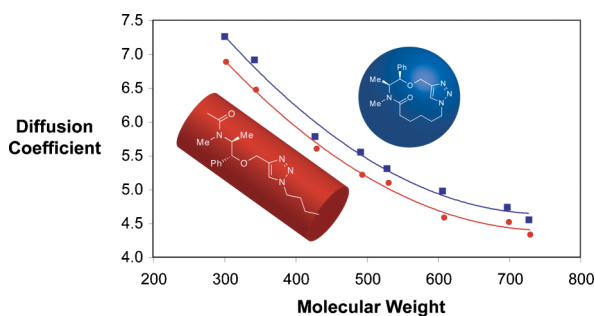


### A heterogeneous Pd–Bi/C catalyst in the synthesis of L-lyxose and L-ribose from naturally occurring D-sugars

Ao Fan,\* Stephan Jaenicke\* and Gaik-Khuan Chuah\*

Green oxidation of sugars to lactones using a heterogeneous Pd–Bi/C catalyst and molecular oxygen.

7727

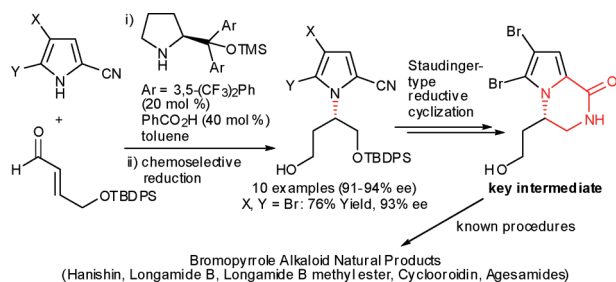


### Comparison of diffusion coefficients for matched pairs of macrocyclic and linear molecules over a drug-like molecular weight range

Andrew R. Bogdan, Nichola L. Davies and Keith James\*

Matched pairs of drug-like macrocycles and acyclic controls have been synthesized and their diffusion behavior compared using NMR spectroscopy.

7734

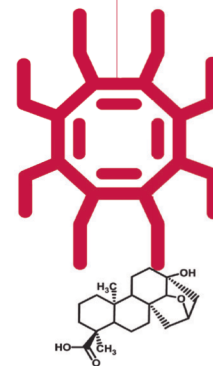
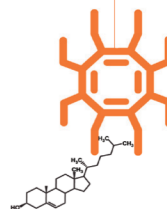
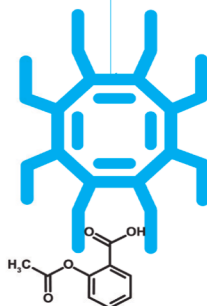
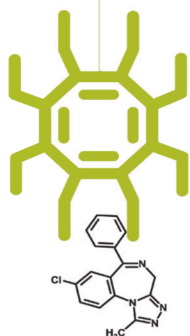
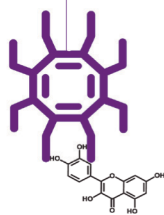


### Organocatalytic enantioselective formal synthesis of bromopyrrole alkaloids via aza-Michael addition

Su-Jeong Lee, Seok-Ho Youn and Chang-Woo Cho\*

The key intermediate of bromopyrrole alkaloids was effectively synthesized via organocatalytic enantioselective aza-Michael addition and a Staudinger-type reductive cyclization.





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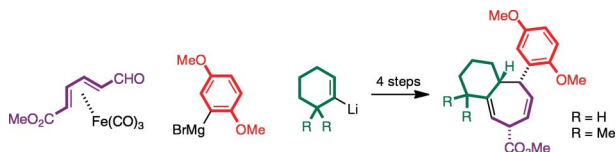
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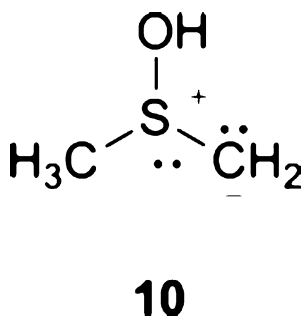


### Reactivity of acyclic (pentadienyl)iron(1+) cations: Synthetic studies directed toward the frondosins

Do W. Lee, Rajesh K. Pandey, Sergey Lindeman and William A. Donaldson\*

A four-step route to the 6-aryl bicyclo[5.4.0]undecane skeleton of the frondosins which relies on the reactivity of (pentadienyl)iron(1+) cations is reported.

7748

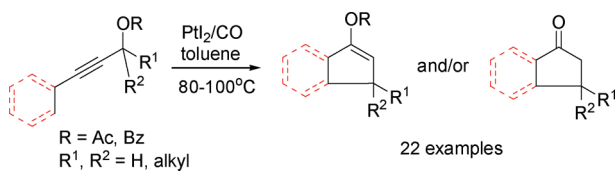


### Concerning the deprotonation of the trimethylsulfonium ion by the dimethylsulfanyl anion

Peter Haiss and Klaus-Peter Zeller\*

The deprotonation of the trimethylsulfonium ion (**1**) by the dimethyl anion (**8**) is accompanied by extensive hydrogen scrambling. The intermediate formation hydroxy-methylsulfonium methylene (**10**) in the acid-base interaction of both **1** and DMSO with **8**, is suggested to account for the hydrogen exchange.

7755

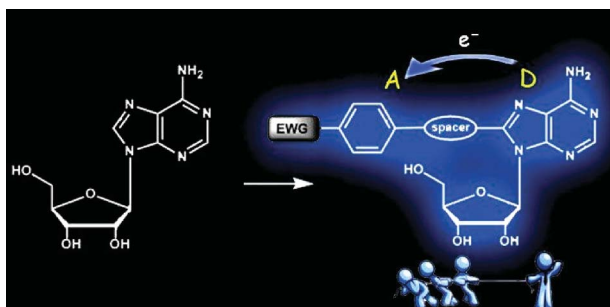


### $\text{PtI}_2$ -Catalyzed tandem 3,3-rearrangement/Nazarov reaction of arylpropargylic esters: synthesis of indanone derivatives

Huaiji Zheng, Xingang Xie, Juan Yang, Changgui Zhao, Peng Jing, Bowen Fang and Xuegong She\*

An efficient  $\text{PtI}_2$ -catalyzed tandem reaction of arylpropargylic esters, involving 3,3-rearrangement and Nazarov reaction, has been developed to produce 3-substituted and 3,3-disubstituted indanone derivatives. This approach provided a pathway to the synthesis of indanone skeletons in natural products.

7763



### 8-(*p*-CF<sub>3</sub>-cinnamyl)-modified purine nucleosides as promising fluorescent probes

Lital Zilbershtein, Alon Silberman and Bilha Fischer\*

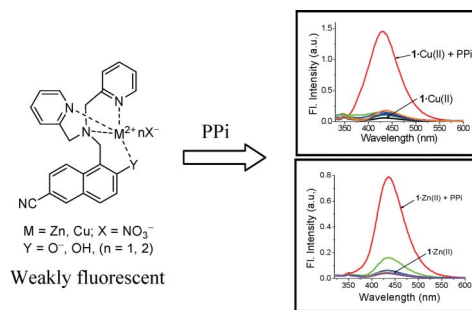
8-(EWG)-modified purine nucleosides proved extremely fluorescent with quantum yield of 0.8 and  $\lambda_{\text{em}}$  of up to 460 nm.

7774

### Mononuclear Zn(II)- and Cu(II)-complexes of a hydroxynaphthalene-derived dipicolylamine: fluorescent sensing behaviours toward pyrophosphate ions

Basab Roy, Alla Sreenivasa Rao and Kyo Han Ahn\*

Two mononuclear Zn(II)-DPA and Cu(II)-DPA complexes crafted on a 2-hydroxy-6-cyanonaphthalene fluorophore are synthesized; they selectively sense pyrophosphates (PPi) over ATP, phosphates, and other anions in aqueous medium, showing 17–24 times fluorescence enhancement. Both the complexes elicit fluorescence increase in a time-dependent fashion.

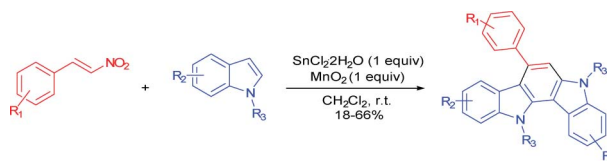


7780

### A one-pot synthesis of 7-phenylindolo[3,2-a]carbazoles from indoles and $\beta$ -nitrostyrenes, *via* an unprecedented reaction sequence

Grégory Dupeyre, Pascale Lemoine, Nabila Ainseba, Sylvie Michel and Xavier Cachet\*

An expeditious synthesis of homodimeric 7-phenylindolo[3,2-a]carbazoles from 1*H*-indoles and  $\beta$ -nitrostyrenes was developed *via* a six-step one-pot reaction. The reactions proceeded under very mild conditions in the presence of the reagent combination  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O} / \text{MnO}_2$ .

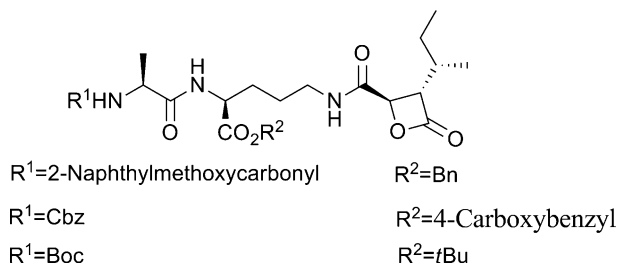


7791

### Synthesis and biological activity of optimized belactosin C congeners

Vadim S. Korotkov, Antje Ludwig, Oleg V. Larionov, Alexander V. Lygin, Michael Groll and Armin de Meijere\*

To understand their structure–activity relations in detail and to find ways of improving their biological activity, four new modified belactosin congeners have been synthesized and tested. One of them turned out to be a more potent inhibitor against HeLa cells than the known proteasome inhibitor MG132.

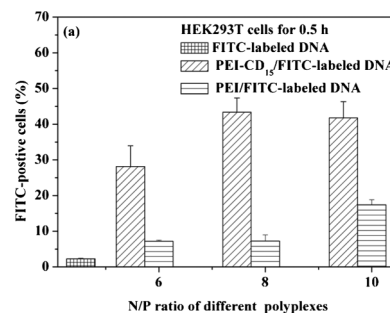


7799

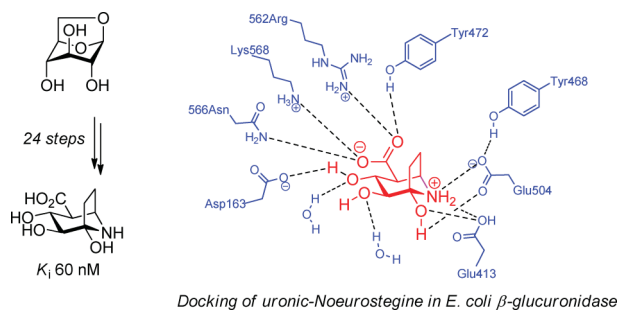
### The influence of cyclodextrin modification on cellular uptake and transfection efficiency of polyplexes

Wenyu Li, Lina Chen, Zhixiong Huang, Xiaofei Wu, Yuanfeng Zhang, Qiaoling Hu and Youxiang Wang\*

The cellular uptake and transfection efficiency of PEI- $\text{CD}_{15}$ /DNA polyplexes were significantly improved by HEK293T cells for the promoted caveolae-mediated endocytosis.



7807

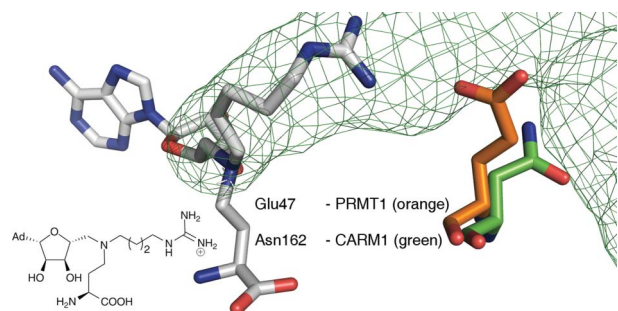


### Synthesis of *uronic*-Noeurostegine – a potent bacterial $\beta$ -glucuronidase inhibitor

Tina S. Rasmussen, Heidi Koldsø, Shinpei Nakagawa, Atsushi Kato, Birgit Schiøtt and Henrik H. Jensen\*

A new, stable hemi-aminal mimic of glucuronic acid could potentially be used in combination with Irinotecan in cancer treatment.

7814

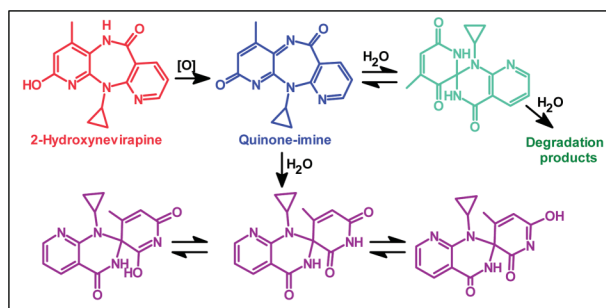


### Small molecule inhibitors that discriminate between protein arginine *N*-methyltransferases PRMT1 and CARM1

James Dowden,\* Richard A. Pike, Richard V. Parry, Wei Hong, Usama A. Muhsen and Stephen G. Ward

One amino acid difference between protein arginine methyltransferases PRMT1 and CARM1 may explain the remarkable selectivity observed for inhibitors characterised herein.

7822

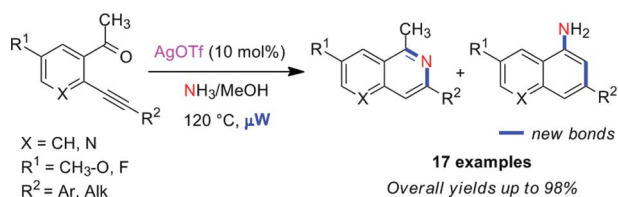


### Synthesis and oxidation of 2-hydroxynevirapine, a metabolite of the HIV reverse transcriptase inhibitor nevirapine

Alexandra M. M. Antunes,\* David A. Novais, J. L. Ferreira da Silva, Pedro P. Santos, M. Conceição Oliveira, Frederick A. Beland and M. Matilde Marques\*

Oxidation of 2-hydroxynevirapine, a metabolite of the anti-HIV drug nevirapine, proceeds through an electrophilic quinone-imine intermediate, which could play a role in nevirapine-induced toxicity.

7836



### Silver-catalysed intramolecular cyclisation of 2-alkynylacetophenones and 3-acetyl-2-alkynylpyridines in the presence of ammonia

Monica Dell'Acqua, Giorgio Abbiati,\* Antonio Arcadi and Elisabetta Rossi

A new silver-catalysed/microwave-assisted approach to 2-methylisoquinolines and 5-methyl-1,6-naphthyridines by tandem addition/cyclisation of  $\gamma$ -ketoalkynes with ammonia.



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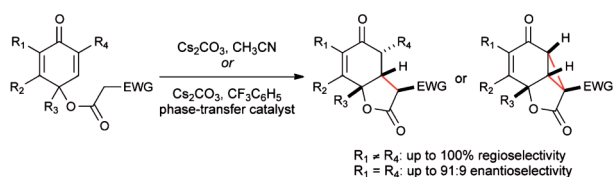
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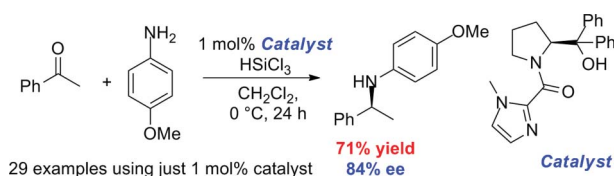


### Regioselective and stereoselective cyclizations of cyclohexadienones tethered to active methylene groups

Rodolfo Tello-Aburto, Kyle A. Kalstabakken, Kelly A. Volp and Andrew M. Harned\*

The ability of steric and electronic influences to control the regioselectivity of cyclizations onto cyclohexadienones has been studied along with the ability of chiral phase-transfer catalysts to desymmetrize achiral cyclohexadienones.

7860



### Scope of the organocatalysed asymmetric reductive amination of ketones with trichlorosilane

François-Moana Gautier, Simon Jones,\* Xianfu Li and Stephen J. Martin

A highly active organocatalyst has been shown to affect the asymmetric reductive amination of ketones producing both aromatic and aliphatic amines. The efficient synthesis of the calcimimetic (+)-NPS R-568 demonstrated the synthetic applicability of this methodology.

7869

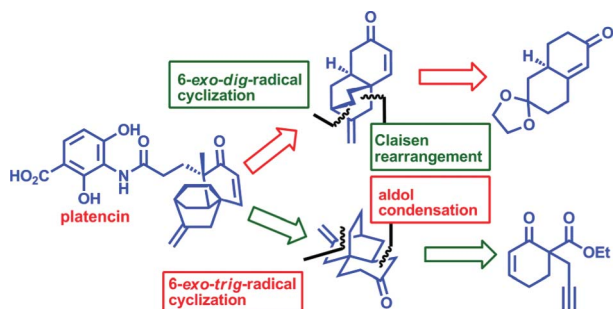


### N-Heterocyclic carbene-catalyzed 1,3-dipolar cycloaddition reactions: a facile synthesis of 3,5-di- and 3,4,5-trisubstituted isoxazoles

Shravankumar Kankala, Ravinder Vadde\* and Chandra Sekhar Vasam\*

A first example of nucleophilic *N*-heterocyclic carbene as catalyst to exploit the click-type fast 1,3-dipolar cycloaddition of nitrile oxide with a variety of alkynes for the regioselective synthesis of desired isoxazoles in high yields is described.

7877



### A radical cyclization approach to the formal total syntheses of platencin

Kalanidhi Palanichamy, Ayyagari V. Subrahmanyam and Krishna P. Kaliappan\*

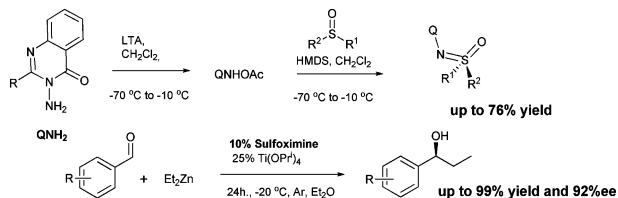
A couple of short and efficient strategies have been developed for the formal synthesis of platencin using Claisen rearrangement, radical cyclization and aldol condensation as key steps with minimal or no use of protective group operations.

7887

### Synthesis of chiral sulfoximines derived from 3-aminoquinazolinones and their catalysis of enantioselective diethylzinc addition to aldehydes

Semistan Karabuga, Murat Cakici, Cavit Kazaz, Ertan Sahin, Hamdullah Kilic and Sabri Ulukanli\*

A series of novel sulfoximines derived from 3-aminoquinazolinone were screened for catalytic enantioselective addition to aromatic aldehydes with a product enantiopurity (ee) of 92%.

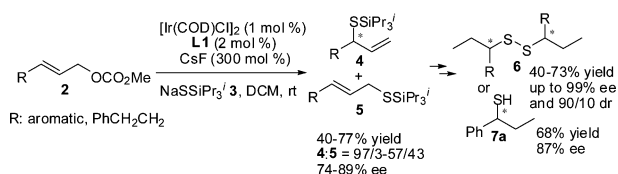


7897

### Iridium-catalyzed asymmetric allylation of sodium triisopropylsulfanethiolate: A new way to form chiral thiols

Weiqing Huang, Shengcai Zheng, Jialiang Tang and Xiaoming Zhao\*

The iridium complex-promoted regio- and enantioselective reaction of allylic carbonates with sodium triisopropylsulfanethiolate produced the branch allylic sulfides which were transformed into chiral thiol or disulfides.

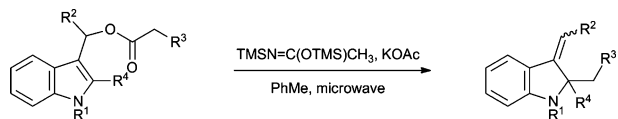


7904

### Decarboxylative Claisen rearrangement reactions: synthesis and reactivity of alkylidene-substituted indolines

Jason E. Camp, Donald Craig,\* Kiyohiko Funai and Andrew J. P. White

Microwave-assisted decarboxylative Claisen rearrangement (dCr) reactions of substituted acetate derivatives of 3-(hydroxyalkyl)indoles give de-aromatized products. The reactivity of the resultant compounds was evaluated.

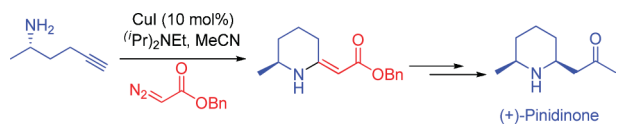


7913

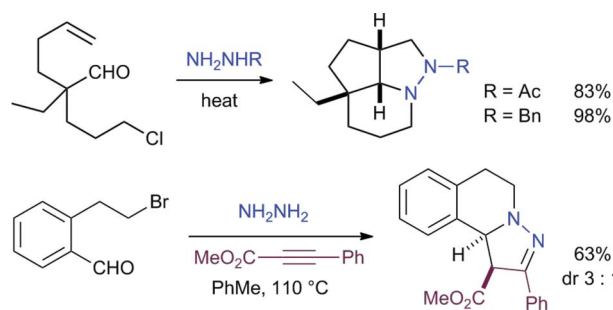
### Cu(I)-catalyzed tandem benzyldiazoester coupling with terminal alkyne–allene formation–Michael reaction: Application to the syntheses of oxa and azacycles

Gullapalli Kumaraswamy,\* Neerasa Jayaprakash and Guniganti Balakishan

A simple and practical procedure for the synthesis of aza- and oxacycles, which possess an array of stereogenic groups, is described.



7921

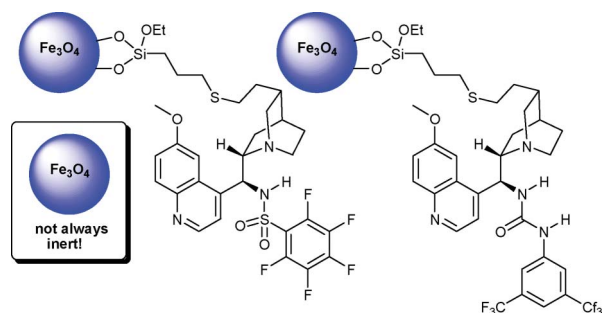


### Cascade cyclization, dipolar cycloaddition of azomethine imines for the synthesis of pyrazolidines

Hélène D. S. Guerrand, Harry Adams and Iain Coldham\*

Heating an aldehyde containing an alkyl halide tether with hydrazines leads to intermediate hydrazones and, after cyclization, azomethine imines that undergo dipolar cycloaddition.

7929

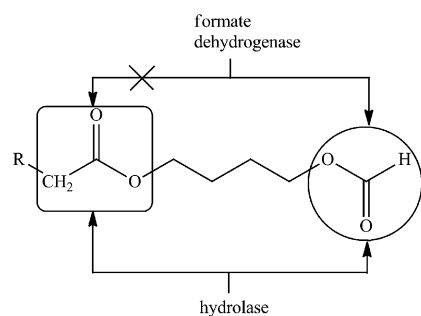


### The immobilisation of chiral organocatalysts on magnetic nanoparticles: the support particle cannot always be considered inert

Oliver Gleeson, Gemma-Louise Davies, Aldo Peschiulli, Renata Tekoriute, Yurii K. Gun'ko\* and Stephen J. Connon\*

The immobilisation of chiral organocatalysts onto magnetic nanoparticles is not always straightforward- the nanoparticle can be more than a spectator.

7941



### Formate dehydrogenase - a biocatalyst with novel applications in organic chemistry

Peter Fröhlich, Kristin Albert and Martin Bertau\*

Formate dehydrogenase affords selective deprotection of formylated alcohols while acylated alcohols remain unaffected.