

Organic & Biomolecular Chemistry

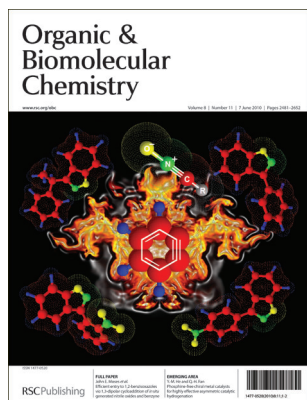
An international journal of synthetic, physical and biomolecular organic chemistry

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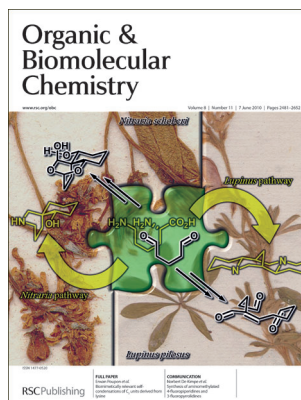
ISSN 1477-0520 CODEN OBCRAK 8(11) 2481–2652 (2010)



Cover

See John E. Moses *et al.*, pp. 2537–2542.
An improved synthesis of a range of 1,2-benzisoxazoles via 1,3-dipolar cycloaddition of *in situ* generated reactive intermediates: nitrile oxides and benzyne is described.

Image reproduced by permission of John E. Moses from *Org. Biomol. Chem.*, 2010, **8**, 2537.



Inside cover

See Erwan Poupon *et al.*, pp. 2522–2528.
Self-condensations of C_3 reactive units enabled the synthesis of skeletons reminiscent to that of alkaloids known to be biosynthesized via the lysine pathway (images of the Linnaeus' herbarium courtesy of the Museum of Natural History, Stockholm, Sweden).

Image reproduced by permission of Erwan Poupon from *Org. Biomol. Chem.*, 2010, **8**, 2522.

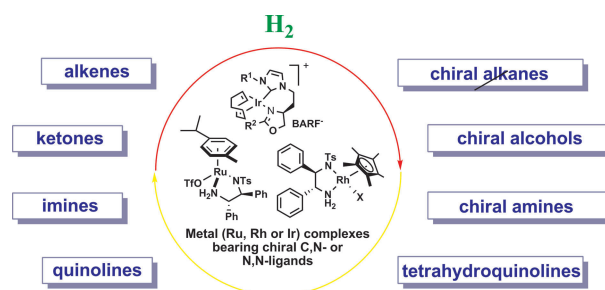
EMERGING AREA

2497

Phosphine-free chiral metal catalysts for highly effective asymmetric catalytic hydrogenation

Yan-Mei He and Qing-Hua Fan*

In this account, two types of chiral phosphine-free ligand, N-heterocyclic carbene-based *C,N*-ligands and diamine-based *N,N*-ligands, in the homogeneous asymmetric hydrogenation of prochiral ketones, imines and quinolines are reviewed.



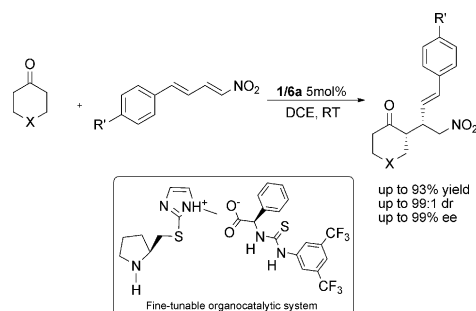
COMMUNICATIONS

2505

The highly enantioselective Michael addition of ketones to nitrodiene catalyzed by the efficient organocatalyst system of pyrrolidinyl-thioimidazole and chiral thioureido acid

Zhao-Bo Li, Shu-Ping Luo, Yi Guo, Ai-Bao Xia and Dan-Qian Xu*

The highly enantioselective asymmetric Michael addition reactions of ketones to nitrodiene was promoted efficiently by fine-tunable organocatalytic system of pyrrolidinyl-thioimidazole and chiral thioureido acid to afford the adducts with high yields (up to 93%), high diastereoselectivities (up to 99 : 1) and excellent enantioselectivities (up to 99% ee).



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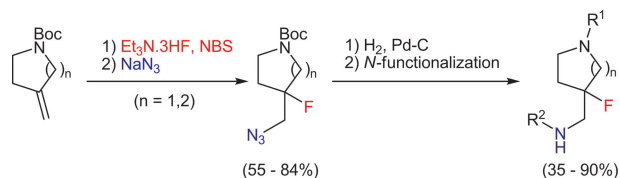
COMMUNICATIONS

2509

Synthesis of aminomethylated 4-fluoropiperidines and 3-fluoropyrrolidines

Guido Verniest, Karel Piron, Eva Van Hende, Jan Willem Thuring, Gregor Macdonald, Frederik Deroose and Norbert De Kimpe*

A short and efficient synthesis of 4-aminomethyl-4-fluoropiperidines and 3-aminomethyl-3-fluoropyrrolidines is described. These fluorinated azaheterocycles are of specific interest as bifunctional building blocks for fluorinated pharmaceutical compounds.

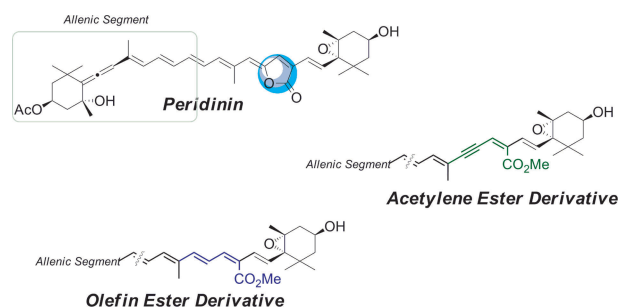


2513

Syntheses of ylidenebutenolide-modified derivatives of peridinin and their stereochemical and spectral characteristics

Takayuki Kajikawa, Kazuyoshi Aoki, Takashi Iwashita, Dariusz M. Niedzwiedzki, Harry A. Frank and Shigeo Katsumura*

We describe the syntheses of two ylidenebutenolide-modified derivatives of peridinin and the results of their stereochemical and spectral characteristics toward elucidation of the exact role of the ylidenebutenolide function.

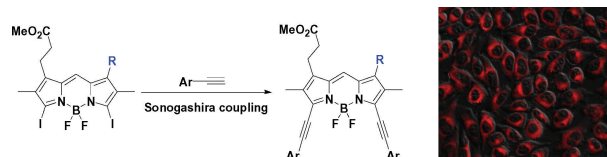


2517

Long wavelength red fluorescent dyes from 3,5-diiodo-BODIPYs

Lijuan Jiao,* Changjiang Yu, Timsy Uppal, Mingming Liu, Yan Li, Yunyou Zhou, Erhong Hao, Xiaoke Hu and M. Graça H. Vicente*

Amphiphilic and long wavelength red fluorescent dyes have been efficiently synthesized from the Sonogashira coupling reactions of 3,5-diiodo-BODIPYs. One of these compounds showed low dark cytotoxicity and accumulated preferentially within the ER of HEP2 cells.

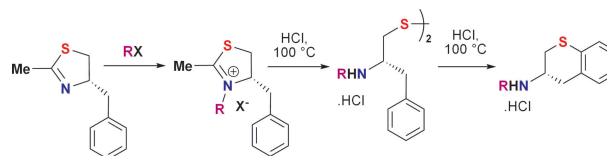


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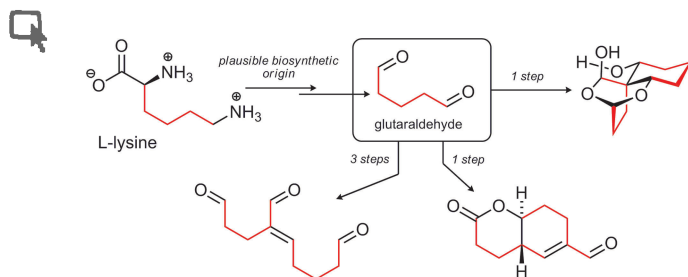
Synthesis of 3-amino-thiochromanes from 4-benzyl 2-thiazolines, via an unprecedented intramolecular electrophilic aromatic substitution

Guillaume Mercey, Remi Legay, Jean-François Lohier, Jana Sopkova-de Oliveira Santos, Jocelyne Levillain, Annie-Claude Gaumont and Mihaela Gulea*

A one-pot synthesis of various *N*-substituted 3-amino-thiochromanes from 4-benzyl-2-methyl thiazoline is described. The reaction involves the formation of a disulfide, which subsequently takes part in an unprecedented intramolecular electrophilic aromatic substitution.



2522

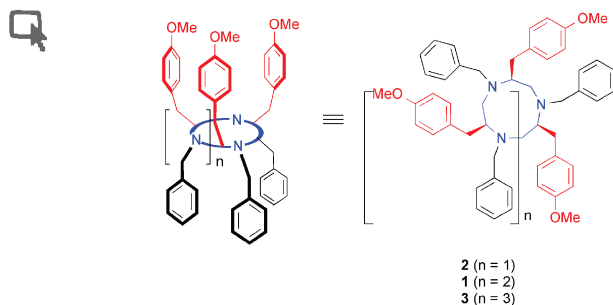


Biomimetically relevant self-condensations of C₅ units derived from lysine

Rim Salame, Edmond Gravel, Pascal Retailleau and Erwan Poupon*

In various and simple conditions, dimerization of pentanedial-derived units gives rise to interesting skeletons, which are reminiscent of alkaloids known to be biosynthesized in Nature *via* lysine metabolism.

2529

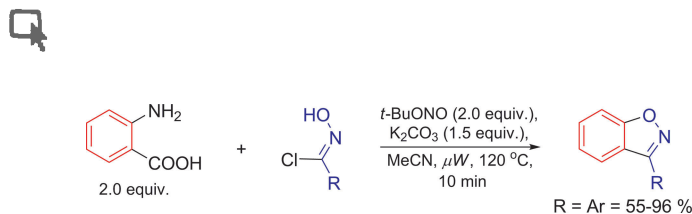


Synthesis of chiral polyazamacrocycles of variable ring size

Seiji Kamioka, Sakae Sugiyama, Takashi Takahashi and Takayuki Doi*

Optically active tri-, tetra-, and penta-azamacrocycles having 4-methoxyphenyl pendants were synthesized. Tri-azamacrocycle **2** does not mainly have a vase-type conformation as tetra-azamacrocycle **1** does but penta-azamacrocycle **3** has a vase-type conformation in CDCl₃ and in CD₂Cl₂.

2537

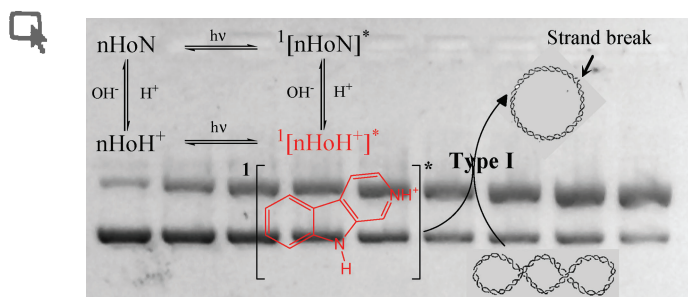


An efficient entry to 1,2-benzisoxazoles *via* 1,3-dipolar cycloaddition of *in situ* generated nitrile oxides and benzyne

Christian Spiteri, Christopher Mason, Fengzhi Zhang, Dougal J. Ritson, Pallavi Sharma, Steve Keeling and John E. Moses*

An efficient protocol for the synthesis of a range of 1,2-benzisoxazoles using an improved 1,3-dipolar cycloaddition of nitrile oxides and benzyne is described. Key to the procedure is the *in situ* generation of the reactive nitrile oxide and benzyne reactants simultaneously.

2543



Photosensitized cleavage of plasmidic DNA by norharmane, a naturally occurring β -carboline

M. Micaela Gonzalez, Magali Pellon-Maison, Matias A. Ales-Gandolfo, Maria R. Gonzalez-Baró, Rosa Erra-Balsells* and Franco M. Cabrerizo*

In air-equilibrated aqueous solution, under both pH conditions, the photosensitized cleavage of plasmidic DNA occurs mainly *via* Type I mechanism (electron transfer) from the single excited state (S₁) of the protonated form of norharmane ($^1[n\text{HoH}^+]^*$).

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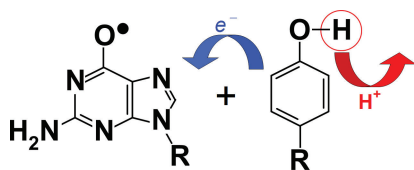
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2553

Reduction of electron deficient guanine radical species in plasmid DNA by tyrosine derivatives

Mandi Tsoi, Trinh T. Do, Vicky J. Tang, Joseph A. Aguilera and Jamie R. Milligan*

Reduction of guanyl radicals in plasmid DNA by tyrosine residues in an electrostatically bound peptide ligand involves a proton coupled electron transfer.

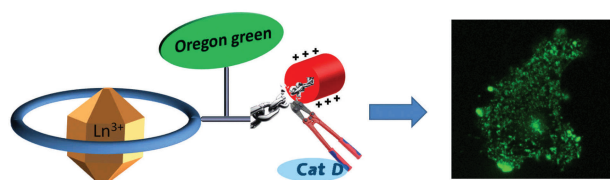


2560

A paramagnetic chemical exchange-based MRI probe metabolized by cathepsin D: design, synthesis and cellular uptake studies

Mojmír Suchý, Robert Ta, Alex X. Li, Filip Wojciechowski, Stephen H. Pasternak, Robert Bartha and Robert H. E. Hudson*

A dual fluorescence/MRI probe for the potential detection of localized cathepsin D activity has been synthesized which includes MRI and optical reporter groups connected to a cell penetrating peptide by a cathepsin D cleavable sequence.

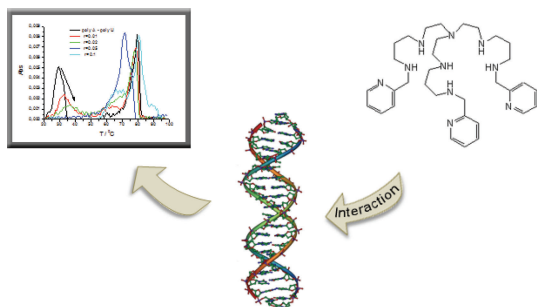


2567

Acid–base properties of functionalised tripodal polyamines and their interaction with nucleotides and nucleic acids

Alejandra Sornosa-Ten, M. Teresa Albelda,* Juan C. Frías, Enrique García-España,* José M. Llinares, Ana Budimir and Ivo Piantanida*

Tripodal polyamines functionalised with heterocyclic moieties revealed the formation of stable complexes with monophosphate nucleotides in aqueous media. Strong binding of all the studied compounds to both ds-DNA and ds-RNA is to some extent selective toward the latter, showing rather rare RNA over DNA preference.

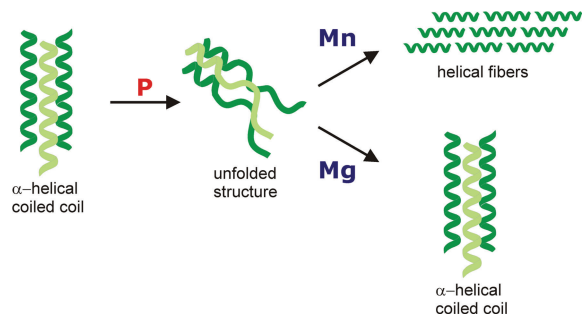


2575

Towards understanding secondary structure transitions: phosphorylation and metal coordination in model peptides

Malgorzata Broncel, Sara C. Wagner, Kerstin Paul, Christian P. R. Hackenberger* and Beate Kokschi*

Structural consequences of phosphorylation and subsequent magnesium and manganese ion coordination were investigated in a coiled coil-based peptide model. It was demonstrated that these biologically relevant factors have significant molecular switching abilities, with phosphorylation being highly destabilizing and metals possessing structure-inducing properties.

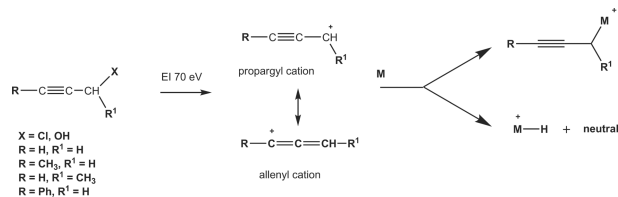


2580

Intrinsic acidity and electrophilicity of gaseous propargyl/allenyl carbocations

Priscila M. Lalli, Yuri E. Corilo, Patricia V. Abdelnur, Marcos N. Eberlin* and Kenneth K. Laali*

The ion/molecule chemistry of propargyl/allenyl cations with different substituents was studied. Their intrinsic acidity, as measured *via* proton transfer reactions, and electrophilicity (adduct formation) were evaluated in reactions with model reactants.

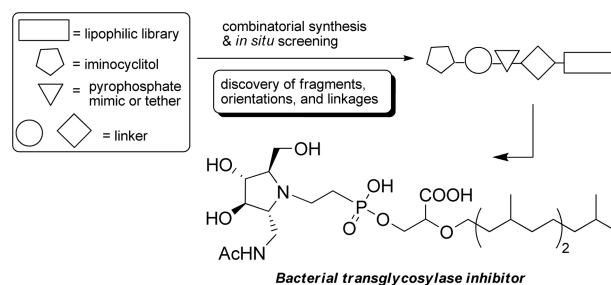


2586

Combinatorial approach toward synthesis of small molecule libraries as bacterial transglycosylase inhibitors

Hao-Wei Shih, Kuo-Ting Chen, Shao-Kang Chen, Chia-Ying Huang, Ting-Jen R Cheng, Che Ma, Chi-Huey Wong* and Wei-Chieh Cheng*

With assistance of microtiter plate-based combinatorial chemistry and *in situ* screening, a potential inhibitor, the first potent iminocyclitol-based inhibitor against bacterial TGases was efficiently developed.

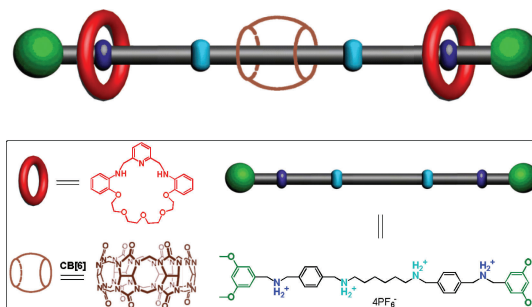


2594

Efficient synthesis of a hetero[4]rotaxane by a “threading-stoppering-followed-by-clipping” approach

Jun Yin, Chunyan Chi and Jishan Wu*

A “threading-stoppering-followed-by-clipping” approach was used for the synthesis of a hetero[4]rotaxane, in which one cucurbit[6]uril (CB[6]) and two hetero crown ether macrocycles are threaded onto one dumbbell-shaped molecule.

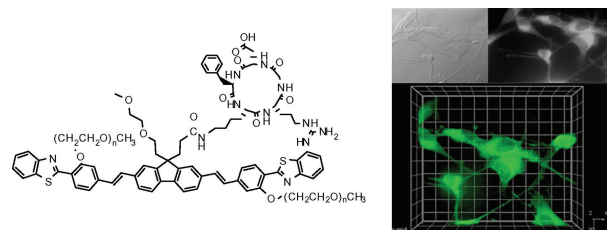


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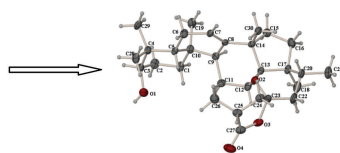
Linear and nonlinear photophysics and bioimaging of an integrin-targeting water-soluble fluorenyl probe

Alma R. Morales, Gheorghe Luchita, Ciceron O. Yanez, Mykhailo V. Bondar, Olga V. Przhonska and Kevin D. Belfield*

A water-soluble fluorenyl probe has been synthesized and its linear photophysical and two-photon absorption properties characterized. Conventional and two-photon fluorescence microscopy imaging of U87MG cells incubated with the fluorenyl-RGD peptide conjugate demonstrated high integrin selectivity.



2609



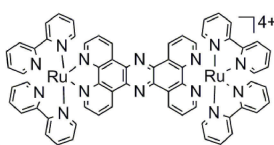
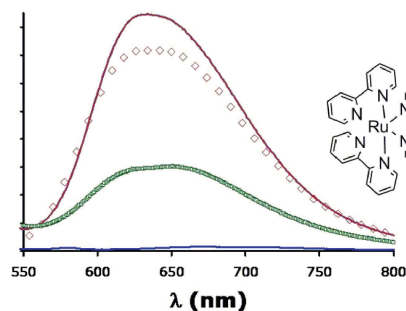
Abiesatrine A (1)

Abiesatrines A–J: anti-inflammatory and antitumor triterpenoids from *Abies georgei* Orr

Xian-Wen Yang, Su-Mei Li, Liang Wu, Yong-Li Li, Lin Feng, Yun-Heng Shen, Jun-Mian Tian, Jian Tang, Ning Wang, Yonghong Liu and Wei-Dong Zhang*

A novel *spiro*-lanostane (abiesatrine A, **1**) was isolated from *Abies georgei* together with 9 new and 10 known triterpenes. The configuration of **1**, featuring a unique spiro-lactone formed by C-13 and C-23 via oxygen-bridge, was confirmed by X-ray crystallography.

2617

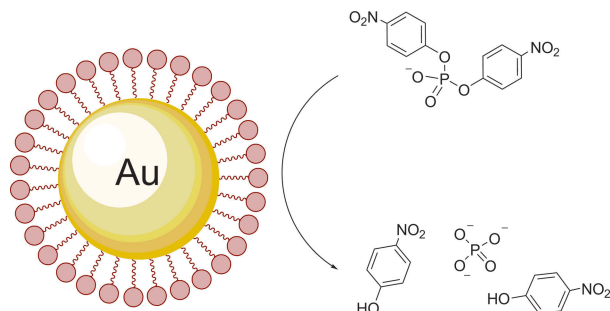


Differentiating quadruplexes: binding preferences of a luminescent dinuclear ruthenium(II) complex with four-stranded DNA structures

Tom Wilson, Mike P. Williamson* and Jim A Thomas*

A DNA dimmer switch: the interaction of dinuclear ruthenium complexes with biologically relevant DNA quadruplexes results in emission that is dependent—in both intensity and wavelength—on specific quadruplex structural features.

2622

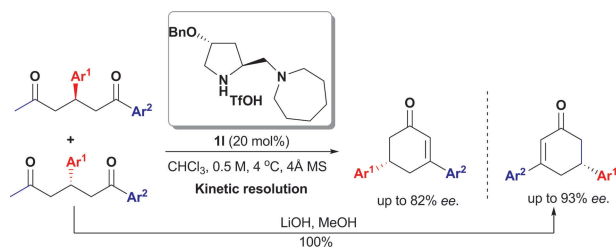


Phosphate diesters cleavage mediated by Ce(IV) complexes self-assembled on gold nanoparticles

Renato Bonomi, Paolo Scrimin and Fabrizio Mancin*

Gold nanoparticles bearing Ce(IV) complexes on the coating monolayer show high activity in the hydrolytic cleavage of phosphate diesters due to the cooperative action of several metal ions.

2627



Organocatalytic kinetic resolution *via* intramolecular aldol reactions: Enantioselective synthesis of both enantiomers of chiral cyclohexenones

Liujuan Chen, Sanzhong Luo,* Jiuyuan Li, Xin Li and Jin-Pei Cheng*

Kinetic resolution of 6-aryl-2,6-hexadiones was achieved with chiral secondary amine catalyzed intramolecular aldolization. The current kinetic resolution protocol enables the synthesis of both enantiomers of cyclohexenones with moderate to good enantioselectivity.

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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*

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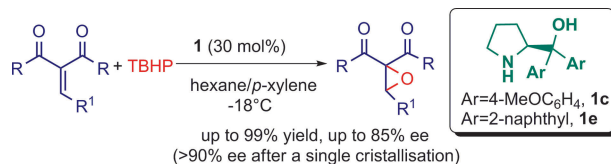
PAPERS

2633


 **Asymmetric epoxidation of 2-arylidene-1,3-diketones: facile access to synthetically useful epoxides**

Alessio Russo and Alessandra Lattanzi*

The first enantioselective epoxidation reaction of acyclic and cyclic 2-arylidene-1,3-diketones, by means of simple α,α -diaryl prolinols/TBHP system, provides the corresponding synthetically and pharmaceutically useful epoxides in up to 85% ee.

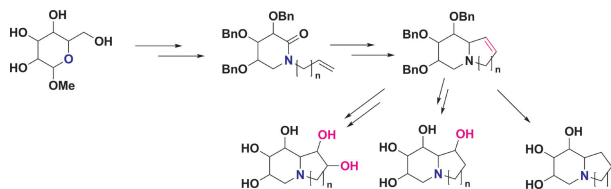


2639

 **A new synthetic access to bicyclic polyhydroxylated alkaloid analogues from pyranosides**


Ning Wang, Li-He Zhang and Xin-Shan Ye*

An efficient route to bicyclic polyhydroxylated alkaloid analogues from pyranosides was developed by using a seven- or eight-step sequence in good overall yields.



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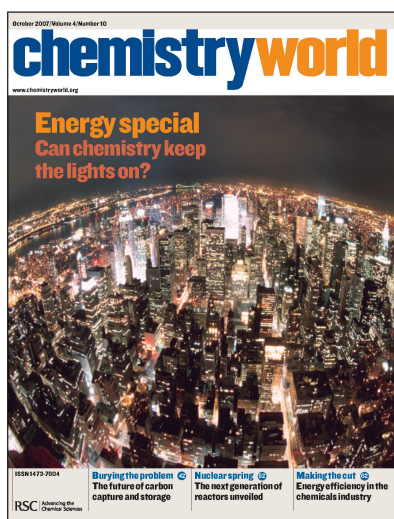
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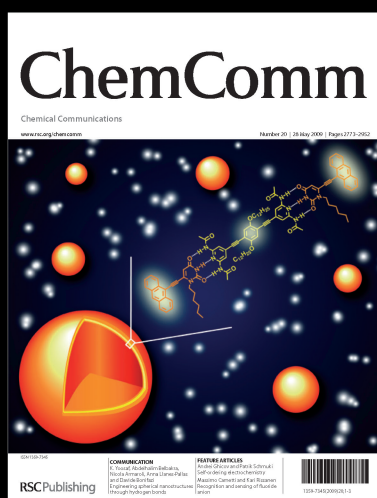
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