

Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

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IN THIS ISSUE

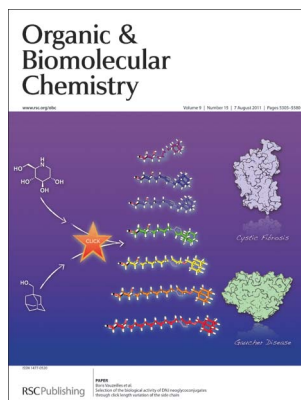
ISSN 1477-0520 CODEN OBCRAK 9(15) 5305–5580 (2011)



Cover

See Asish K. Bhattacharya and co-workers, pp. 5407–5413.

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Inside cover

See Boris Vauzeilles *et al.*, pp. 5373–5388.

Click-chemistry allows easy modulation of the biological activity of deoxynojirimycin-derived neoglycoconjugates (protein structures are from the pdb; F508del-CFTR, 1xmj, and glucocerebrosidase, 2v3f).

Image reproduced by permission of Boris Vauzeilles from *Org. Biomol. Chem.*, 2011, **9**, 5373.

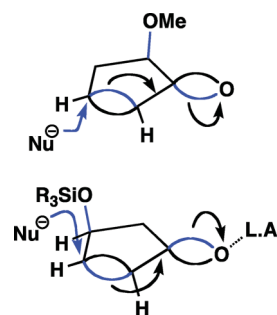
PERSPECTIVE

5321

Bent bonds, the antiperiplanar hypothesis and the theory of resonance. A simple model to understand reactivity in organic chemistry

Ghislain Deslongchamps* and Pierre Deslongchamps*

Bent bonds in action: A fresh look at the bent bond model for unsaturated systems in conjunction with modern stereoelectronic principles reveals a wide range of applicability to the understanding of conformation, reactivity, and stereochemistry.



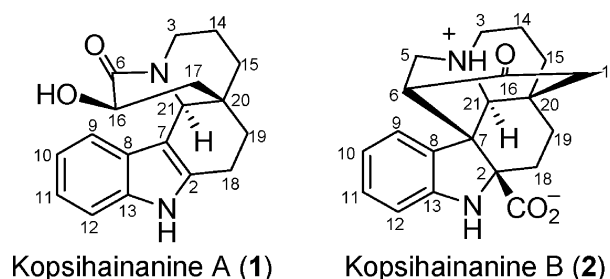
COMMUNICATIONS

5334

Kopsihainanines A and B, two unusual alkaloids from *Kopsia hainanensis*

Jia Chen, Jian-Jun Chen, Xiaojun Yao and Kun Gao*

Kopsihainanine A, possessing an unprecedented skeleton with a pentacyclic rearranged ring system, and kopsihainanine B, a new zwitterionic alkaloid, were reported.



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Organic & Biomolecular Chemistry (print: ISSN 1477-0520; electronic: ISSN 1477-0539) is published 24 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF.

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Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

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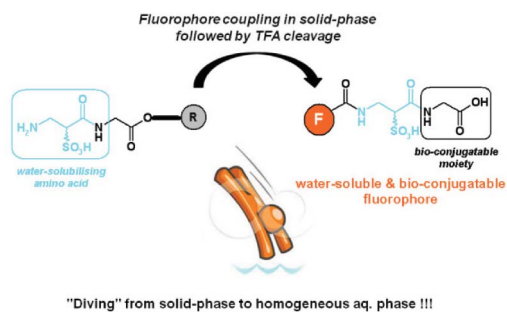
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5337

N-Fmoc- α -sulfo- β -alanine: a versatile building block for the water solubilisation of chromophores and fluorophores by solid-phase strategy

Anthony Romieu,* Thomas Bruckdorfer, Guillaume Clavé, Virgile Grandclaude, Cédrik Massif and Pierre-Yves Renard

The use of sulfonated amino acid α -sulfo- β -alanine in standard Fmoc SPPS has been described for the first time. Application of this “on resin” sulfonation strategy to the rapid synthesis of water-soluble chromophores/fluorophores is presented.

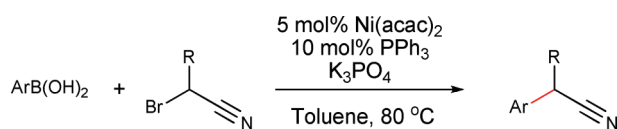


5343

Novel α -arylnitriles synthesis *via* Ni-catalyzed cross-coupling of α -bromonitriles with arylboronic acids under mild conditions

Yingying Yang, Shan Tang, Chao Liu, Huimin Zhang, Zhexun Sun and Aiwen Lei*

An applicable and easy-handling Ni-catalyst can be used to promote direct arylation of α -bromonitriles with various arylboronic acids to construct α -arylnitriles under mild conditions. The methodology tolerates β -hydrogens and functional groups in the substrates.

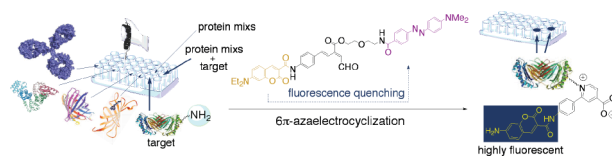


5346

Target-selective fluorescent “switch-on” protein labeling by 6π -azaelectrocyclization

Katsunori Tanaka,* Masataka Kitadani and Koichi Fukase*

Application of azaelectrocyclization and FRET techniques to lysine groups enables selective and sensitive detection of target proteins with high fluorescence contrast.

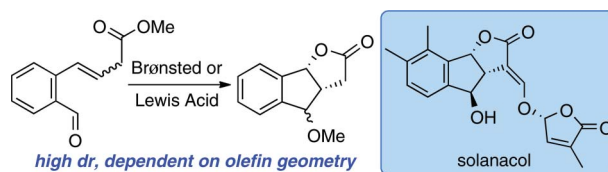


5350

Synthetic studies on the solanacol ABC ring system by cation-initiated cascade cyclization: implications for strigolactone biosynthesis

Kinga Chojnacka, Stefano Santoro, Radi Awartani, Nigel G. J. Richards, Fahmi Himo and Aaron Aponick*

A highly selective acid-catalyzed method for construction of the ABC ring system of strigolactones from simple precursors is reported.

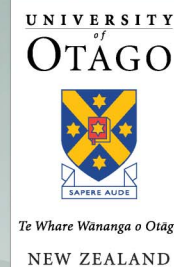




29 Jan – 2 Feb, 2012, Dunedin, New Zealand

OTAGO

2012 ISMSC-7



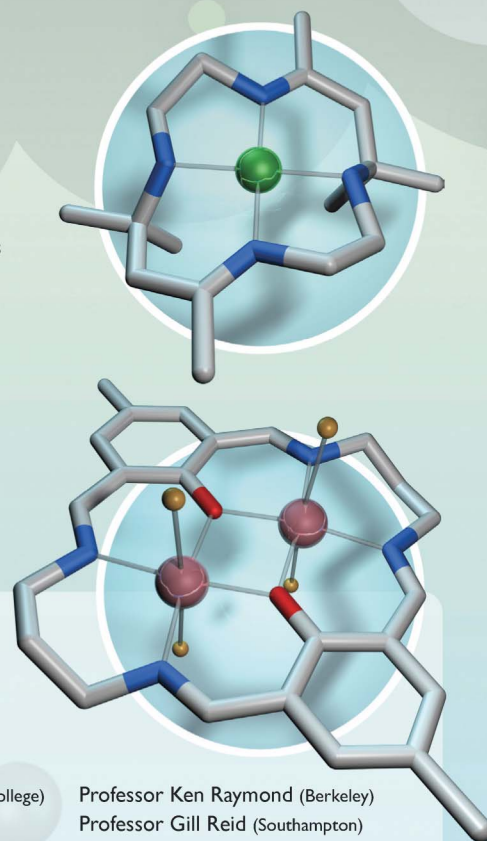
It is with great pleasure that we extend an invitation to you to join us at the University of Otago in Dunedin, in our summer, for the **2012 International Symposium on Macrocyclic and Supramolecular Chemistry, 29 January – 2 February 2012.**

Dunedin and Otago Peninsula are renowned for their beauty. Dunedin is also a gateway to the beautiful scenery of Central Otago, the Catlins and Stewart Island.

Majestic Victorian and Edwardian architecture, a proximity to an abundance of wildlife and a healthy youth culture will make for an exciting and fascinating visit.

We have an impressive list of invited speakers lined up and are confident that attendees will find their trip to the far south, "downunder", both valuable and rewarding.

We promise you a lively and invigorating scientific programme – so why not come and join us!
– Professors Sally Brooker & Keith Gordon



Confirmed plenary public lecture:

Professor Sir Fraser Stoddart (Northwestern)

Confirmed invited keynote speakers to date:

Professor Paul Beer (Oxford)
Professor Terry Collins (Carnegie Mellon)
Professor Tony Davis (Bristol)
Professor Luisa de Cola (Munster)
Professor Sylvia Draper (Trinity Dublin)
Professor Kim Dunbar (Texas A&M)
Professor Makoto Fujita (Tokyo)
Professor Phil Gale (Southampton)
Professor Juan Granja (Santiago de Compostela)

Professor Thorri Gunnlaugsson (Trinity College)
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Professor Alan Rowan (Nijmegen)
Professor Jonathan Sessler (Texas)
Professor Hanadi Sleiman (McGill)
Professor Jonathan Steed (Durham)
Professor Michael Ward (Sheffield)
Professor Vivian Yam (Hong Kong)

N.B. The list of confirmed invited keynote lecturers will be updated periodically so please keep an eye on the conference website.



For further information and to register your interest please go to our conference website:

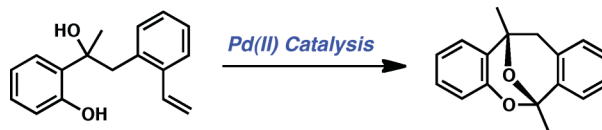
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5354

A rapid and convergent synthesis of the integrastatin core

Pamela M. Tadross, Pradeep Bugga and Brian M. Stoltz*

The [3.3.1]-dioxabicyclic core of the integrastatin natural products has been prepared by a Pd(II)-catalyzed oxidative cyclization in only 4 linear steps from known compounds.

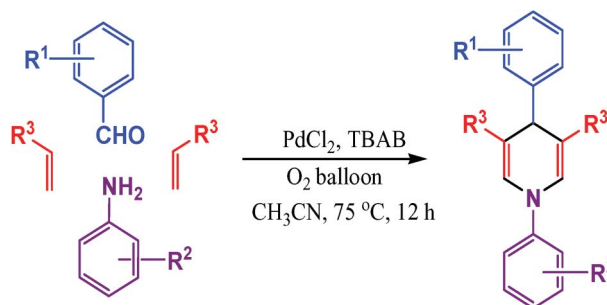


5358

Palladium-assisted multicomponent cyclization of aromatic aldehydes, arylamines and terminal olefins under molecular oxygen: an assembly of 1,4-dihydropyridines

Huanfeng Jiang,* Xiaochen Ji, Yibiao Li, Zhengwang Chen and Azhong Wang

We describe a novel protocol for the synthesis of 2,6-unsubstituted 1,4-dihydropyridines (1,4-DHPs) *via* palladium-assisted one-pot three-component reactions using molecular oxygen as a sole oxidant.

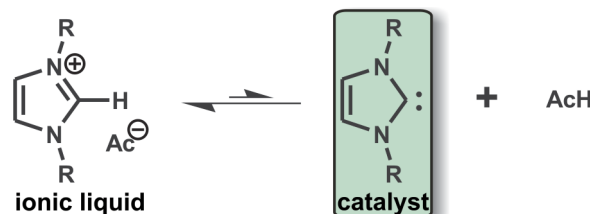


5362

An organocatalytic ionic liquid

Zsolt Kelemen, Oldamur Hollóczy, József Nagy and László Nyulászi*

The carbene concentration in 1-ethyl-3-methylimidazolium-acetate ionic liquid is sufficiently high to act as a catalyst in benzoin condensation, hydroacylation and also in oxidation of an alcohol by using CO₂ and air. This observation reveals the potential of ionic liquid organocatalysts, uniting the beneficial properties of these two families of compounds.

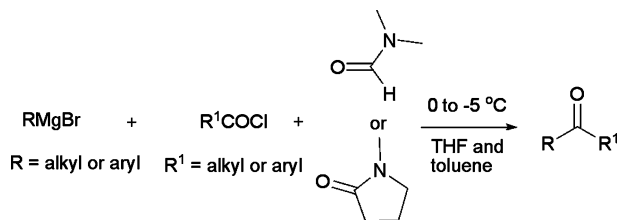


5365

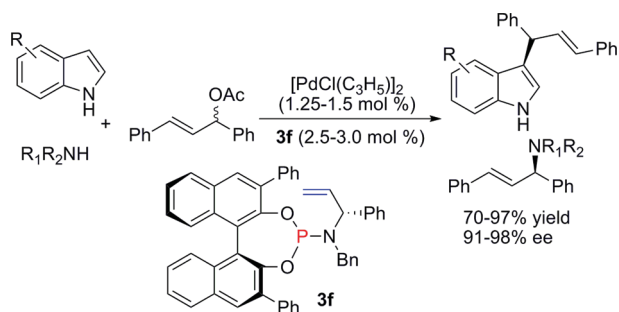
Acylation of Grignard reagents mediated by N-methylpyrrolidone: A remarkable selectivity for the synthesis of ketones

Maravanhalli Sidde Gowda, Sushanth Sudhir Pande, Ramesha Andagar Ramakrishna* and Kandikere Ramaiah Prabhu*

An efficient user-friendly method of acylation of Grignard reagents to selectively synthesize ketones is presented, which is assisted by simple amides such as NMP, or DMF.



5369



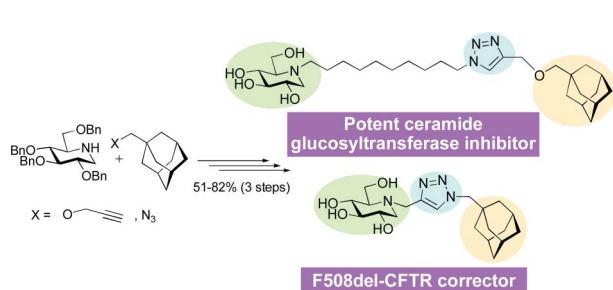
Highly effective chiral phosphorus amidite-olefin ligands for palladium-catalyzed asymmetric allylic substitutions

Zhaoqun Liu, Ziping Cao and Haifeng Du*

Novel P-olefin hybrid ligands have been successfully developed for palladium-catalyzed asymmetric allylic alkylations to furnish high yields with excellent ee's.

PAPERS

5373

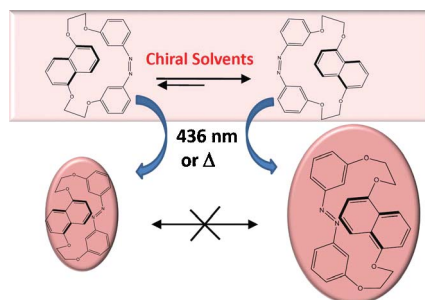


Selection of the biological activity of DNJ neoglycoconjugates through click length variation of the side chain

N. Ardes-Guisot, D. S. Alonzi, G. Reinkensmeier, T. D. Butters, C. Norez, F. Becq, Y. Shimada, S. Nakagawa, A. Kato, Y. Blériot,* M. Sollogoub and B. Vauzeilles*

Click connection between functionalised deoxyjirimycins and adamantanes allows easy tuning of the biological activity profile of the resulting neoglycoconjugates.

5389

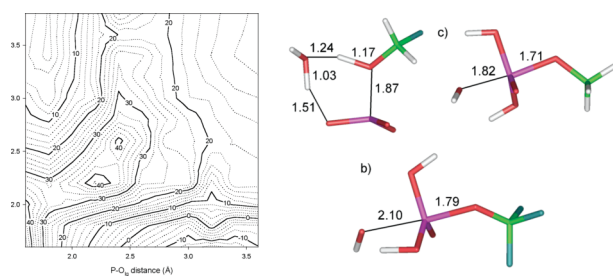


Chirality transfer from chiral solvents and its memory in an azobenzene derivative exhibiting photo-switchable racemization

Reji Thomas and Nobuyuki Tamaoki*

Chirality is transferred from a chiral solvent and memorized as a well defined planar chirality of the single small molecule.

5394



The effect of leaving group on mechanistic preference in phosphate monoester hydrolysis

Shina C. L. Kamerlin and John Wilkie*

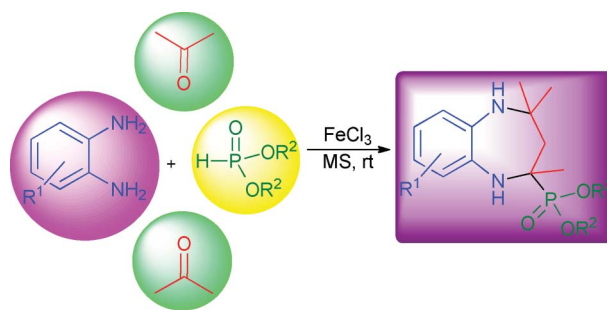
Increasing leaving group stability in phosphate monoester hydrolysis leads to progressive favouring of a dissociative (D_N + A_N) mechanism in small model systems, while for the most stable leaving group, the associative mechanism switches from a step-wise (A_N + D_N) mechanism to a concerted (A_ND_N) mechanism.

5407

An efficient synthesis of benzodiazepinyl phosphonates as clostripain inhibitors *via* FeCl₃ catalyzed four-component reaction

Asish K. Bhattacharya,* Kalpeshkumar C. Rana, Dnyaneshwar S. Raut, Vaibhav P. Mhaindarkar and Mohamad I. Khan

New MCR for the synthesis of benzodiazepinyl phosphonates (BDPs) has been developed by FeCl₃-catalyzed four-component condensation of diamines, acetone and phosphites. The synthesized BDPs have shown significant protease inhibition activity against clostripain.

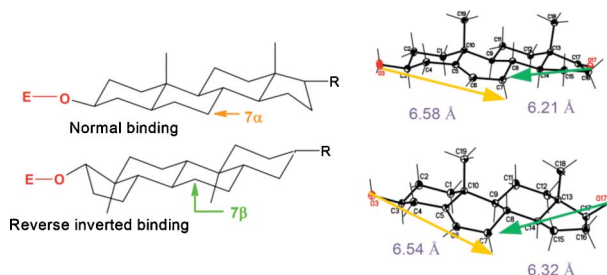


5414

Hydroxylation of DHEA, androstenediol and epiandrosterone by *Mortierella isabellina* AM212. Evidence indicating that both constitutive and inducible hydroxylases catalyze 7 α - as well as 7 β -hydroxylations of 5-ene substrates

Teresa Kołek, Natalia Milecka, Alina Świzdor,* Anna Panek and Agata Białońska

With respect to the oxidating center of the enzyme, the 7 α -H and 7 β -H are located in analogous positions.

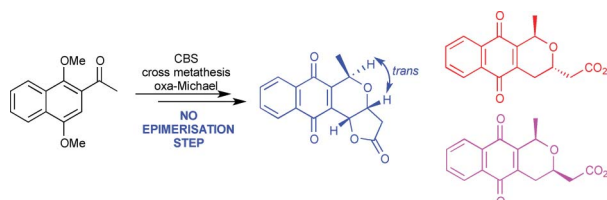


5423

Enantioselective synthesis of pyranonaphthoquinone antibiotics using a CBS reduction/cross-metathesis/oxa-Michael strategy

Paul A. Hume, Jonathan Sperry and Margaret A. Brimble*

A novel synthetic approach delivers naphthopyrans possessing the natural *trans*-stereochemistry observed in the pyranonaphthoquinone family of antibiotics.

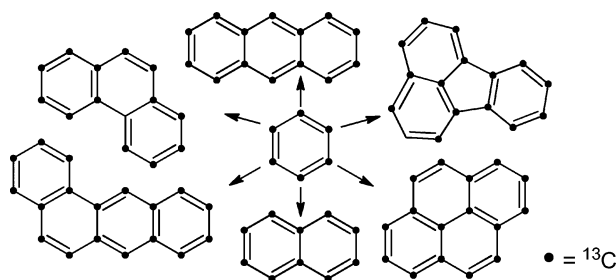


5431

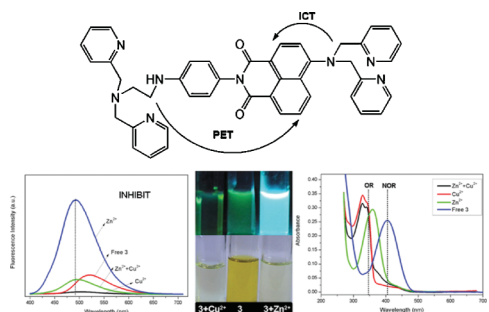
Synthesis of uniformly ¹³C-labeled polycyclic aromatic hydrocarbons

Zhenfa Zhang, Ramiah Sangaiah, Avram Gold* and Louise M. Ball*

Convergent pathways were devised for U-¹³C PAH synthesis *de novo* starting from U-¹³C-benzene and simple, commercially-available ¹³C-compounds.



5436

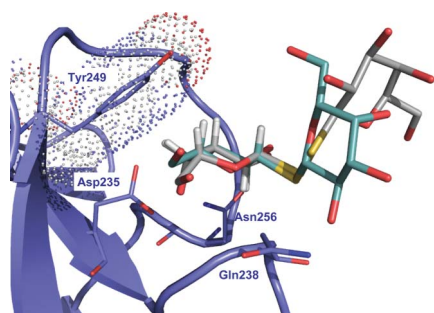


A naphthalimide fluorophore with efficient intramolecular PET and ICT Processes: Application in molecular logic

Haixia Wang, Haixia Wu, Lin Xue, Yan Shi and Xiyou Li*

A novel 4-amino-1,8-naphthalimide with two different cation receptors can selectively coordinate with Zn^{2+} and/or Cu^{2+} and achieve several logic operations.

5445

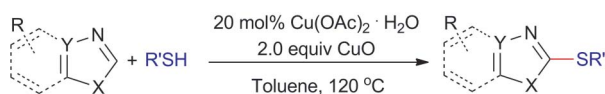


Symmetric dithiodigalactoside: strategic combination of binding studies and detection of selectivity between a plant toxin and human lectins

S. Martín-Santamaría, S. André, E. Buzamet, R. Carballo, G. Fernández-Cureses, M. Morando, J. P. Ribeiro, K. Ramírez-Gualito, B. de Pascual-Teresa, F. J. Cañada, M. Menéndez, O. Ramström, J. Jiménez-Barbero,* D. Solís and H.-J. Gabius

Studies show glycosyldisulfides have potential as chemical platform for inhibitor design.

5456



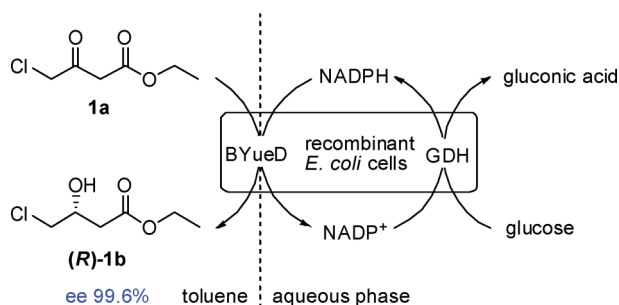
X = O, S, NCH_3 , NBn
 Y = C, N
 R = H, CH_3 , OCH_3 , Cl, NO_2
 R' = Alkyl, Aryl

Copper-catalyzed direct thiolation of azoles with aliphatic thiols

An-Xi Zhou, Xue-Yuan Liu,* Ke Yang, Shu-Chun Zhao and Yong-Min Liang

Copper-catalyzed direct thiolation of azoles with aliphatic thiols is described *via* intermolecular C–S bond formation/C–H functionalization.

5463



Highly stereoselective reduction of prochiral ketones by a bacterial reductase coupled with cofactor regeneration

Yan Ni, Chun-Xiu Li,* Li-Juan Wang, Jie Zhang and Jian-He Xu*

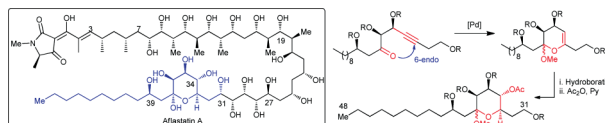
An efficient “tailor-made” whole cell biocatalyst was developed for the highly enantioselective reduction of prochiral ketones.

5469

A [Pd]-mediated ω -alkynone cycloisomerization approach for the central tetrahydropyran unit and the synthesis of C(31)–C(48) fragment of aflastatin A

Sachin B. Narute, Neella Chandra Kiran and Chepuri V. Ramana*

A combination of alkynone cycloisomerization and hydroboration–oxidation addressed the construction of the central *manno*-configured *C*-pyranoside core of aflastatin A.

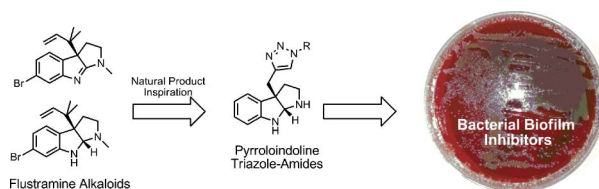


5476

Flustramine inspired synthesis and biological evaluation of pyrroloindoline triazole amides as novel inhibitors of bacterial biofilms

Cynthia Bunders, John Cavanagh and Christian Melander*

Anti-biofilm agents based upon the flustramine family of alkaloids isolated from *Flustra foliacea* have been developed.

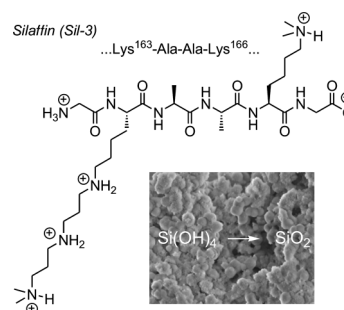


5482

Silica precipitation with synthetic silaffin peptides

Ralph Wieneke, Anja Bernecker, Radostan Riedel, Manfred Sumper, Claudia Steinem* and Armin Geyer*

Different morphologies of precipitating silica are generated by silaffin-derived peptides which contain propyleneamine substituted lysine residues.

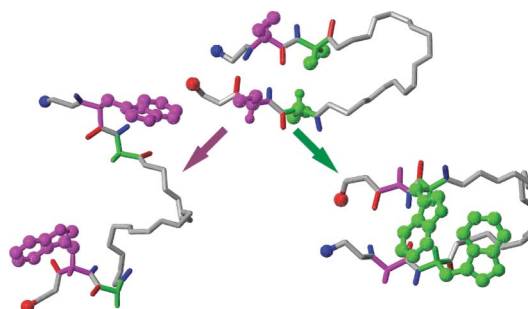


5487

Trp–Trp pairs as β -hairpin stabilisers: Hydrogen-bonded versus non-hydrogen-bonded sites

Clara M. Santiveri, María Jesús Pérez de Vega, Rosario González-Muñiz and M. Angeles Jiménez*

The β -hairpin-stabilising ability of Trp–Trp pairs at non-hydrogen-bonded sites is lost at hydrogen-bonded sites.



1st PACN Congress on Agricultural Productivity

How the chemical sciences can help to feed the world

21 – 23 November 2011

Accra International Convention Centre, Accra, Ghana



The Royal Society of Chemistry (RSC) is joining together with Syngenta to host the first Pan Africa Chemistry Network (PACN) Congress on Agricultural Productivity. This innovative conference will bring leading scientists together to discuss current research and ideas in the agricultural sciences - helping to feed the world!

Themes

Topics at the congress will include:

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- Plant Nutrition
- Soil and Ecosystems
- Innovation in Agricultural Practices
- Adaptation to Climate Change

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19 September 2011

Registration deadline:

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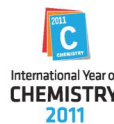
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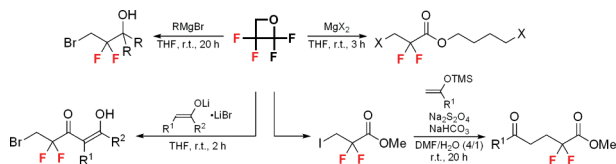
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5493

Easy access to CF₂-containing molecules based on the reaction of 2,2,3,3-tetrafluorooxetane with various nucleophiles

Shigeyuki Yamada, Masahiro Kato, Yudai Komori, Tsutomu Konno and Takashi Ishihara*

Synthesis of various CF₂-containing molecules based on the nucleophilic reaction with 2,2,3,3-tetrafluorooxetane, is reported.

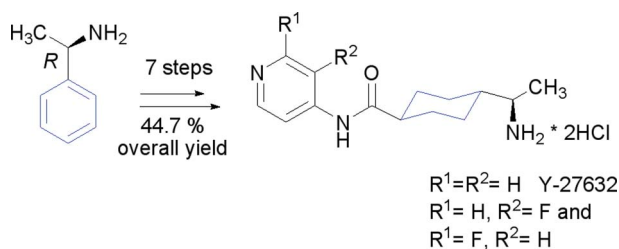


5503

A practical synthesis of Rho-Kinase inhibitor Y-27632 and fluoro derivatives and their evaluation in human pluripotent stem cells

Jiří Paleček, Robert Zweigerdt, Ruth Olmer, Ulrich Martin, Andreas Kirschning* and Gerald Dräger*

Compared to Y-27632 the new fluoro derivatives showed reduced or no effect on hPSC vitality and expansion after dissociation in human pluripotent stem cells.

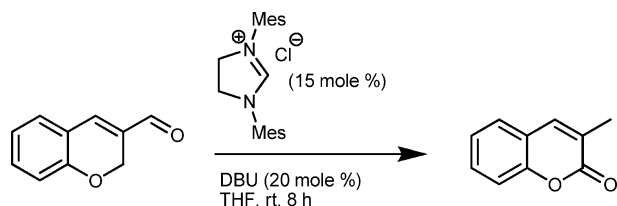


5511

A novel NHC-catalyzed transformation of 2H-chromene-3-carboxaldehydes to 3-methyl-2H-chromen-2-ones

Vijay Nair,* C. R. Sinu, R. Rejithamol, K. C. Seetha Lakshmi and Eringathodi Suresh

An unexpected transformation of 2H-chromene-3-carboxaldehydes to coumarin derivatives, mediated by NHC, is reported.

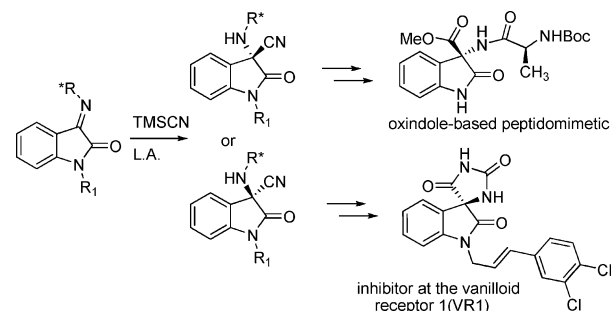


5515

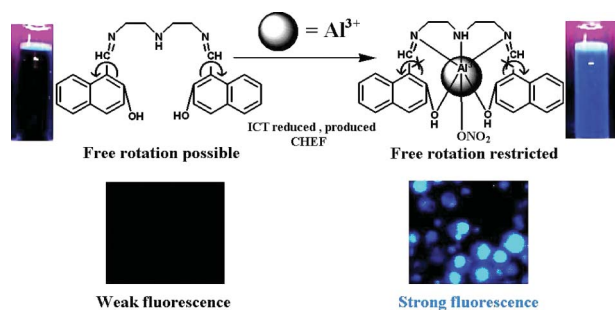
Addition of TMSCN to chiral ketimines derived from isatin. Synthesis of an oxindole-based peptidomimetic and a bioactive spirohydantoin

Alessandro Sacchetti,* Alessandra Silvani,* Francesco G. Gatti, Giordano Lesma, Tullio Pilati and Beatrice Trucchi

The Strecker-type reaction of isatin derived chiral ketimines afforded optically active α -amino nitriles, versatile intermediates for medicinal chemistry applications.



5523

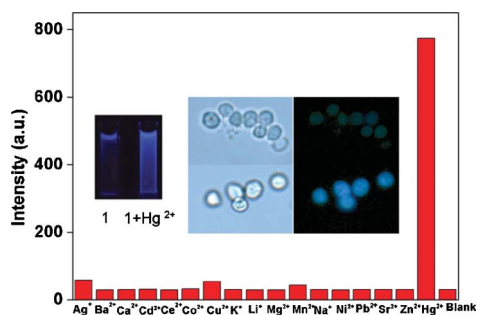


A naphthalene-based Al³⁺ selective fluorescent sensor for living cell imaging

Animesh Sahana, Arnab Banerjee, Sudipta Das, Sisir Lohar, Debasis Karak, Bidisha Sarkar, Subhra Kanti Mukhopadhyay, Asok K. Mukherjee* and Debasis Das*

An efficient Al³⁺ selective naphthalene-based fluorescent probe (L) capable of imaging intracellular Al³⁺ in living cells at physiological pH range (7–7.6) is reported.

5530

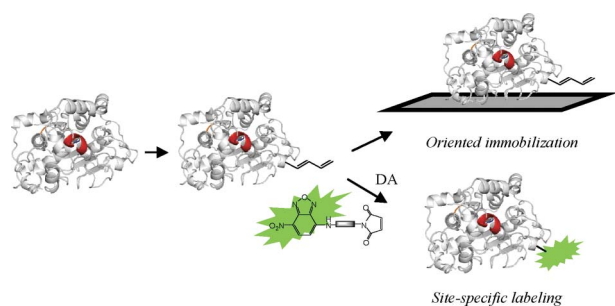


Naphthylthiourea-modified permethylcyclodextrin as a highly sensitive and selective “turn-on” fluorescent chemosensor for Hg²⁺ in water and living cells

Yong Chen, Zhan-Hu Sun, Bao-E Song and Yu Liu*

A naphthylthiourea-modified cyclodextrin showed highly sensitive and selective fluorescence sensing to Hg²⁺ in both water and living cells.

5535

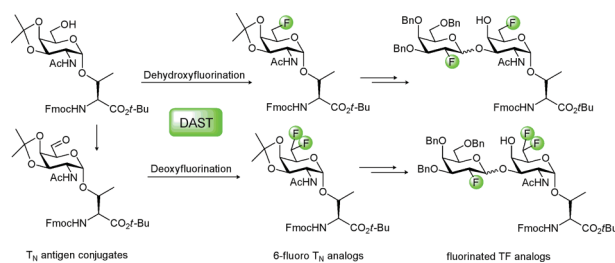


trans,trans-2,4-Hexadiene incorporation on enzymes for site-specific immobilization and fluorescent labeling

Marco Filice, Oscar Romero, Jose M. Guisan and Jose M. Palomo*

Chemical incorporation of a hexadiene moiety on an enzyme molecule permitted protein immobilization and site-specific labeling with NBD fluorophore by Diels–Alder reaction.

5541



Synthesis of fluorinated Thomsen–Friedenreich antigens: direct deoxyfluorination of αGalNAc-threonine *tert*-butyl esters

Manuel Johannes, Thomas Oberbillig and Anja Hoffmann-Röder*

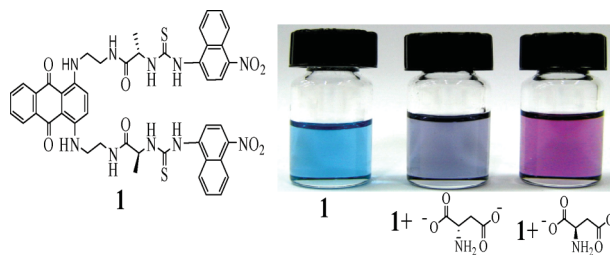
Selectively fluorinated analogs of the tumor-associated T_N and Thomsen–Friedenreich antigens can be efficiently prepared *via* direct DAST-mediated de(hydr)oxyfluorination of orthogonally protected Fmoc-Thr(αGalNAc)-*O**t*Bu building blocks.

5547

Synthesis of alanine-based colorimetric sensors and enantioselective recognition of aspartate and malate anions

Wei-Chi Lin, Yu-Ping Tseng, Chi-Yung Lin and Yao-Pin Yen*

Two new chiral colorimetric sensors were synthesised and they showed distinct color changes when treated with chiral dicarboxylate anions (D/L-aspartate and D/L-malate). Thus, they can act as optical chemosensors for enantioselective discrimination between D/L-aspartate and D/L-malate anions.

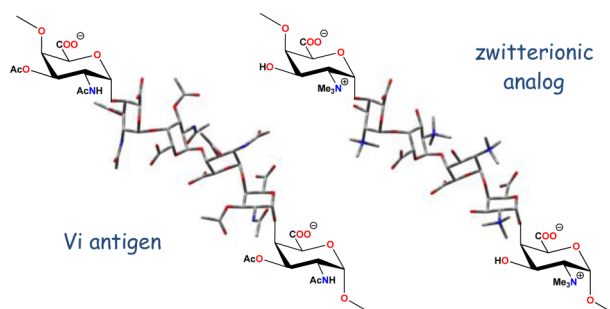


5554

Molecular dynamics simulations of the *Salmonella typhi* Vi antigenic polysaccharide and effects of the introduction of a zwitterionic motif

Laura Legnani, Federica Compostella, Giovanni Grazioso, Franca Marinone Albini and Lucio Toma*

Hexasaccharides corresponding to the Vi capsular antigen and to analogs containing a zwitterionic motif have been modeled through MD simulations.

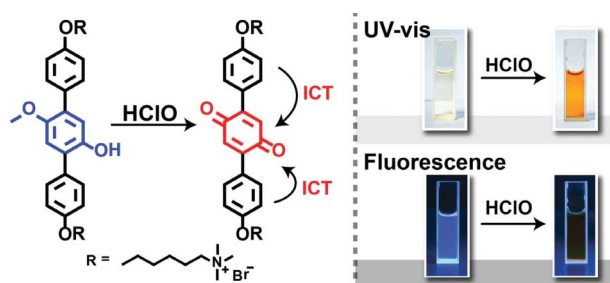


5560

Naked-eye visible and fluorometric dual-signaling chemodosimeter for hypochlorous acid based on water-soluble *p*-methoxyphenol derivative

Wenjun Zhang, Chi Guo, Lihan Liu, Jingui Qin and Chuluo Yang*

A naked-eye visible and fluorometric dual-signaling chemodosimeter for HClO is developed by the oxidation of a simple *p*-methoxyphenol derivative, which induces an intramolecular charge transfer from the phenyl units to the benzoquinone. This detection can be run in aqueous solution with high selectivity over other reactive oxygen species.

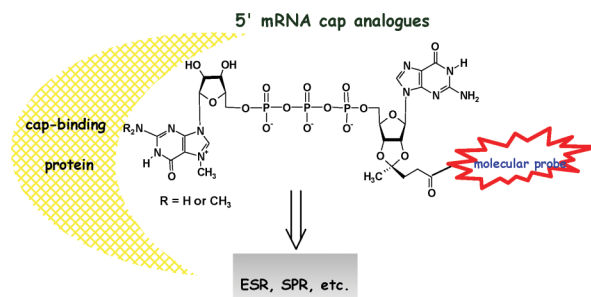


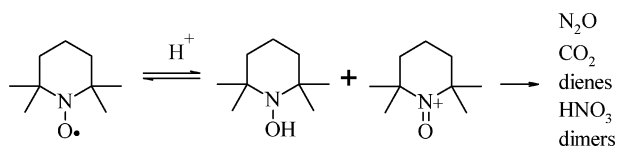
5564

Synthesis of a new class of ribose functionalized dinucleotide cap analogues for biophysical studies on interaction of cap-binding proteins with the 5' end of mRNA

Marzena Jankowska-Anyszka,* Karolina Piecyk and Jelena Šamonina-Kosicka

Herein, we report the synthesis of novel ribose functionalized dinucleotide cap analogues with different introduced molecular probes (biotin, EDA and TEMPO) designed for biophysical studies on the interaction of cap-binding proteins with the 5' end of mRNA.





Thermal decay of TEMPO in acidic media *via* an *N*-oxoammonium salt intermediate

Yun Ma, Colin Loyns, Peter Price and Victor Chechik*

Thermal decay of TEMPO in acids proceeds *via* an *N*-oxoammonium salt intermediate. The main product (*ca.* 80%) is the corresponding hydroxylamine.

A Healthy, Wealthy, Sustainable World

John Emsley



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John Emsley

Foreword by C David Garner

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