

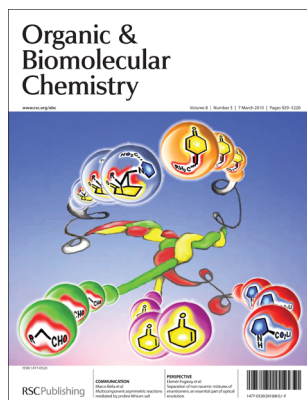
Organic & Biomolecular Chemistry

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

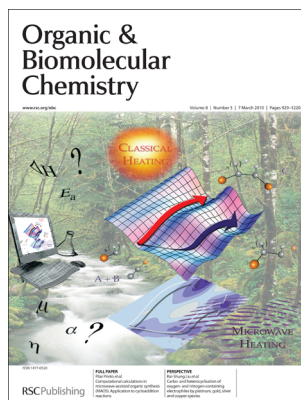
ISSN 1477-0520 CODEN OBCRAK 8(5) 929–1220 (2010)



Cover

See Marco Bella *et al.*, pp. 980–983.
 Bicyclic adducts bearing five stereocenters and novel fragrances are produced by the multicomponent reaction between proline lithium salt, aliphatic aldehydes and 2-cyclohexen-1-one. Authors thank Miss Susy Piovesana for designing this cover.

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Inside cover

See Pilar Prieto *et al.*, pp. 1000–1009.
 The occurrence of thermal and non-thermal effects of microwave irradiation in some reactions could be studied using computational calculations.

Image reproduced by permission of A. de Cózar, M. C. Millán, C. Cebrián, P. Prieto, A. Díaz-Ortiz, A. de la Hoz and F. P. Cossío from *Org. Biomol. Chem.*, 2010, **8**, 1000.

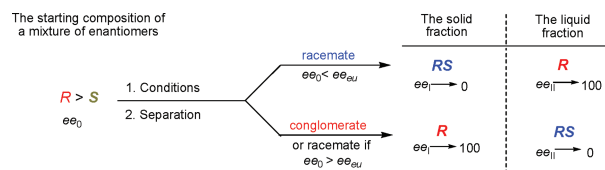
PERSPECTIVES

947

Separation of non-racemic mixtures of enantiomers: an essential part of optical resolution

Ferenc Faigl, Elemér Fogassy,* Mihály Nógrádi, Emese Pálovics and József Schindler

Enrichment of non-racemic mixtures of enantiomers is an important part of resolution processes. All purification methods are based on the racemate- or conglomerate-like behaviour of enantiomers. In this compilation we review the most often used and some throughout uncommon methods based on momentous recognitions.

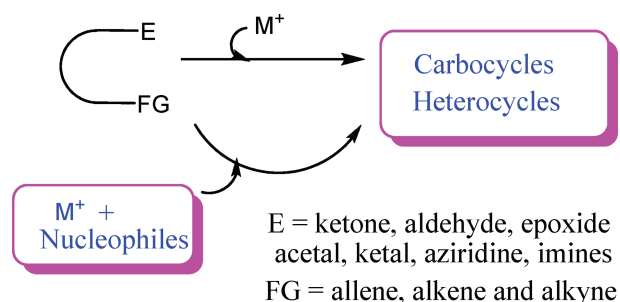


960

Carbo- and heterocyclisation of oxygen- and nitrogen-containing electrophiles by platinum, gold, silver and copper species

Arindam Das, Shariar Md. Abu Sohel and Rai-Shung Liu*

In this present perspective, we summarise the recent progress on the use of gold, platinum, silver and copper complexes to activate common oxygen and nitrogen electrophiles.



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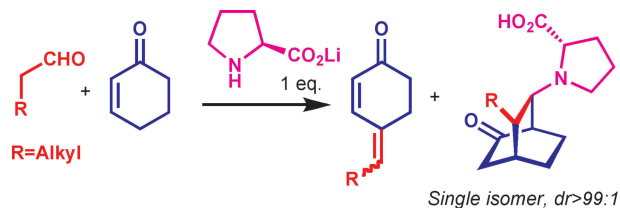
COMMUNICATIONS

980

Multicomponent asymmetric reactions mediated by proline lithium salt

Polysyena Renzi, Jacob Overgaard and Marco Bella*

Bicyclic adducts bearing five stereocenters and novel fragrances are produced by the multicomponent reaction between proline lithium salt, aliphatic aldehydes and 2-cyclohexen-1-one.

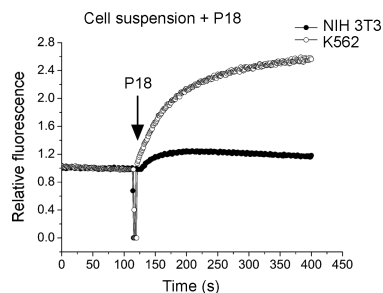


984

Anticancer mechanism of peptide P18 in human leukemia K562 cells

Chengkang Tang, Ximing Shao, Binbin Sun, Wenli Huang, Feng Qiu, Yongzhu Chen, Ying-kang Shi, Er-yong Zhang, Chen Wang and Xiaojun Zhao*

Studies on the anticancer mechanism of peptide P18 in human leukemia K562 cells revealed that P18 causes the K562 cell death by depolarizing plasma membrane potential and enhancing membrane permeability, rather than activating the classical apoptosis pathway.

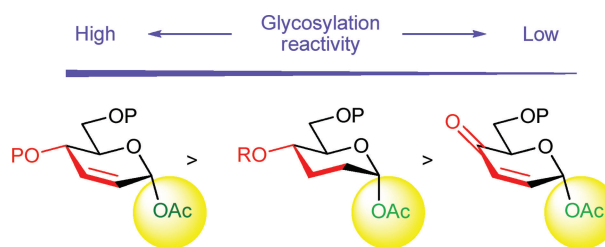


988

Chemoselective glycosylations using 2,3-unsaturated-4-keto glycosyl donors

Shunichi Kusumi, Sainan Wang, Tatsuya Watanabe, Kaname Sasaki, Daisuke Takahashi and Kazunobu Toshima*

Chemoselective glycosylations were effectively performed using 2,3-unsaturated glycosyl and 2,3-dideoxy glycosyl acetates as armed glycosyl donors, and 2,3-unsaturated-4-keto glycosyl acetates as disarmed glycosyl donors.

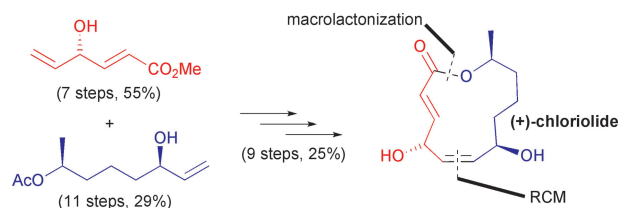


991

Total synthesis of (+)-chloriolide

Timm T. Haug and Stefan F. Kirsch*

The first total synthesis of (+)-chloriolide, a 12-membered macrolide from *Chloridium virescens* (var. *chlamydosporum*), was accomplished in a longest linear sequence of 20 steps from commercial materials in 7% overall yield.



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

13 - 15 September 2010
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Call for posters now open

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.

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*The University of British Columbia,
Canada*

William D. Jones
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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
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Offers of contributed papers related to the listed themes for poster presentation are invited by 16 July 2010.
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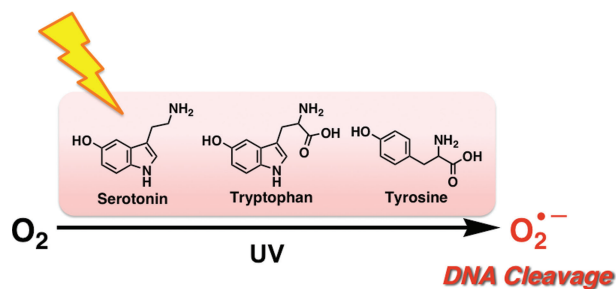
COMMUNICATIONS

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 **Photoinduced DNA cleavage by formation of ROS from oxygen with a neurotransmitter and aromatic amino acids**

Tomonori Kawashima, Kei Ohkubo and Shunichi Fukuzumi*

UV-B photoirradiation of serotonin, tryptophan and tyrosine with oxygen results in DNA cleavage by generation of reactive oxygen species as demonstrated by agarose gel electrophoresis with pBR 322 DNA.

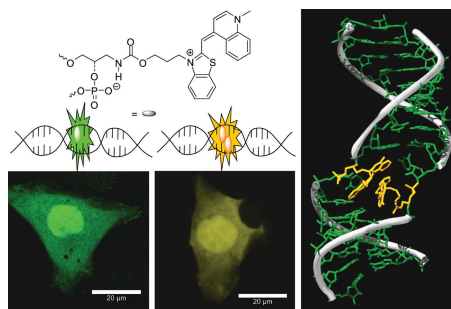


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 **Imaging of RNA delivery to cells by thiazole orange as a fluorescent RNA base substitution**


Sina Berndt, Miriam Breunig, Achim Göpferich and Hans-Achim Wagenknecht*

A fluorescent chameleon for RNA imaging: interstrand thiazole orange dimers in RNA show a yellow-colored emission that can be distinguished from the green TO monomer emission in RNA by confocal microscopy.



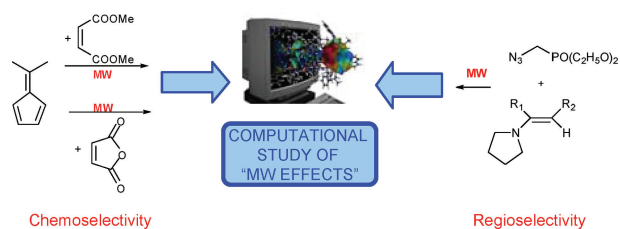
PAPERS

1000

 **Computational calculations in microwave-assisted organic synthesis (MAOS). Application to cycloaddition reactions**

A. de Cózar, M. C. Millán, C. Cebrián, P. Prieto,* A. Díaz-Ortiz,* A. de la Hoz and F. P. Cossío

A DFT computational study of two pericyclic reactions is reported. The computational calculations represent a very useful tool to study separately the occurrence of thermal and non-thermal effects of microwave irradiation.

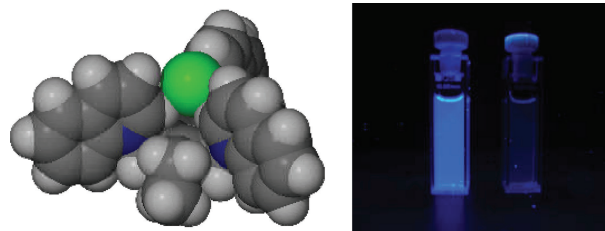


1010

 **A quinolinium-derived turn-off fluorescent anion sensor**

Adam N. Swinburne, Martin J. Paterson, Andrew Beeby* and Jonathan W. Steed*

A quinolinium-derived anion sensor has been synthesised which shows a turn-off fluorescence response in the presence of anions, with selectivity for acetate. The compound exhibits complex anion binding comprising of a host dimer, 2 : 1 and 1 : 1 host : guest species.





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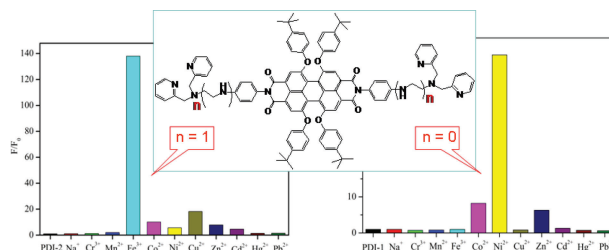
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1017

Nickel(II) and iron(III) selective off-on-type fluorescence probes based on perylene tetracarboxylic diimide

Haixia Wang, Delou Wang, Qi Wang, Xiyou Li* and Christoph A. Schalley*

Two novel fluorescent probes based on perylene tetracarboxylic diimide (PDI) with turn-on output have been prepared. Because of the different linkers between the receptor and the fluorophore, the selectivity of the probes is significantly altered.

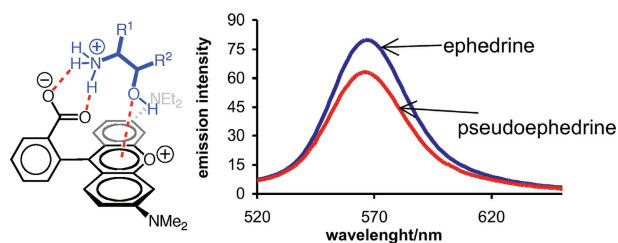


1027

A fluorescent diastereoselective molecular sensor for 1,2-aminoalcohols based on the rhodamine B lactone-zwitterion equilibrium

Clifton J. Stephenson and Ken D. Shimizu*

Rhodamine dye was shown to be able to differentiate and measure the diastereoselectivity of 1,2-aminoalcohols by monitoring the fluorescence of the zwitterionic form.

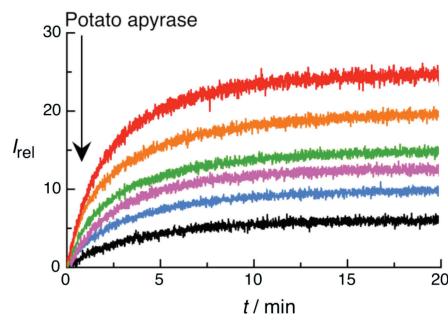


1033

Implementation of anion-receptor macrocycles in supramolecular tandem assays for enzymes involving nucleotides as substrates, products, and cofactors

Mara Florea and Werner M. Nau*

Anion-receptor macrocycles, in combination with fluorescent dyes, can be exploited for the kinetic monitoring of the activity of nucleotide triphosphate-dependent enzymes such as apyrases.

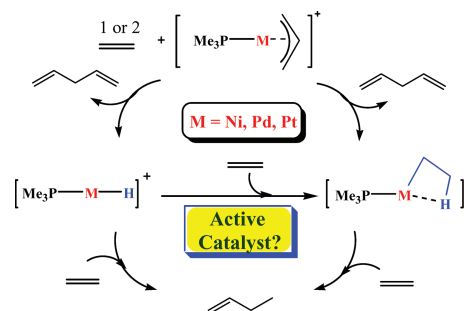


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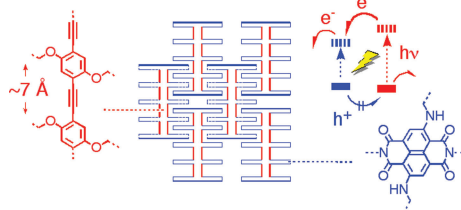
Ni-, Pd-, or Pt-catalyzed ethylene dimerization: a mechanistic description of the catalytic cycle and the active species

Dipankar Roy and Raghavan B. Sunoj*

Mechanistic insights on ethylene dimerization by using $[M(\eta_3\text{-allyl})(PMe_3)]^+$, where $M = Ni(II), Pd(II), \text{ and } Pt(II)$, are presented. The computed DFT energies have been employed to propose the likely nature of the 'active catalyst' in the catalytic cycle.



1052

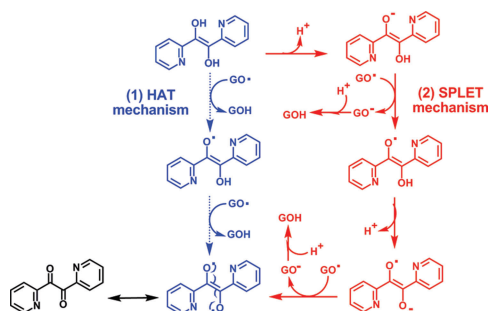


Optoelectronically mismatched oligophenylethynyl-naphthalenediimide SHJ architectures

Santanu Maity, Rajesh Bhosale, Natalie Banerji, Eric Vauthey, Naomi Sakai* and Stefan Matile*

Components of synthetic organic photosystems that are not integrated into supramolecular *n/p*-heterojunctions are shown to generate weak photocurrents only, whereas SHJ-compatible components are operational in the same system.

1058

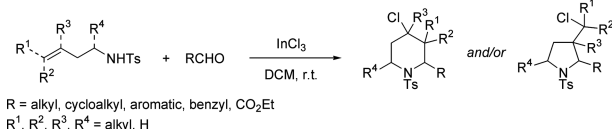


Antioxidant activity of α -pyridoin and its derivatives: possible mechanism

Li-Xia Cheng, Xiao-Ling Jin, Qing-Feng Teng, Jin Chang, Xiao-Jun Yao, Fang Dai,* Yi-Ping Qian, Jiang-Jiang Tang, Xiu-Zhuang Li and Bo Zhou*

This work demonstrates that α -pyridoin and its derivatives are effective antioxidants, and the hydrogen atom transfer (HAT) and sequential proton loss electron transfer (SPLET) mechanisms are responsible for antioxidant reaction.

1064

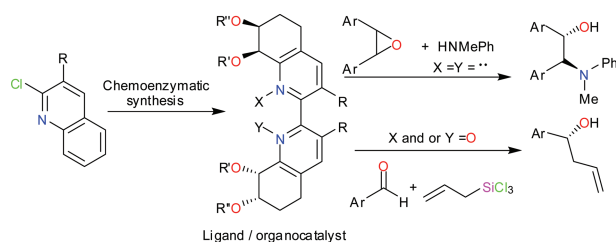


A detailed investigation of the aza-Prins reaction

Adrian P. Dobbs,* Sebastien J. J. Guesné, Robert J. Parker, John Skidmore, Richard A. Stephenson and Mike B. Hursthouse

Indium trichloride has been found to be a highly successful and mild Lewis acid for promoting the aza-Prins reaction, and a thorough mechanistic investigation is described.

1081



Chemoenzymatic synthesis of chiral 2,2'-bipyridine ligands and their *N*-oxide derivatives: applications in the asymmetric aminolysis of epoxides and asymmetric allylation of aldehydes

D. R. Boyd,* N. D. Sharma, L. Sbircea, D. Murphy, J. F. Malone, S. L. James, C. C. R. Allen and J. T. G. Hamilton

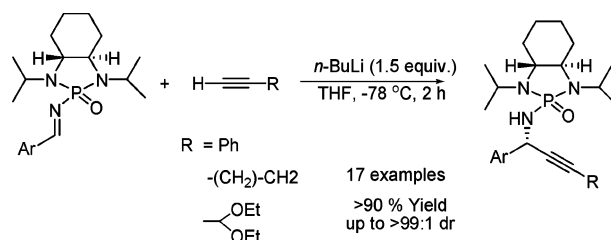
Enantiopure 2,2'-bipyridine *N*-oxides, derived from 2-chloroquinolines, are used in the asymmetric aminolysis of *meso*-epoxides and the asymmetric allylation of aldehydes

1091

Chiral *N*-phosphonyl imine chemistry: an efficient asymmetric synthesis of chiral *N*-phosphonyl propargylamines

Parminder Kaur, Gaurav Shakya, Hao Sun, Yi Pan* and Guigen Li*

Chiral *N*-phosphonylimines were reacted with lithium acetylides to give substituted chiral propargylamines. The types of bases for generating acetylides and solvents are crucial for effectiveness of this asymmetric reaction. Seventeen examples were studied to give excellent yields (>90%) and diastereoselectivities (>96:4 to >99:1).

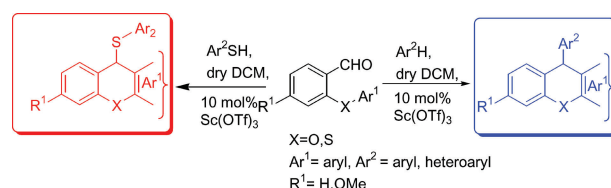


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Scandium triflate-catalyzed one-pot domino approach towards general and efficient syntheses of unsymmetrical 9-substituted xanthenes derivatives

Ritesh Singh and Gautam Panda*

A general and efficient one-pot cascade/tandem approach to synthesize unsymmetrical 9-aryl/heteroaryl xanthenes as well as 9-(thioaryl) xanthenes has been developed under extremely mild reaction conditions using 10 mol% Sc(OTf)₃ as a catalyst.

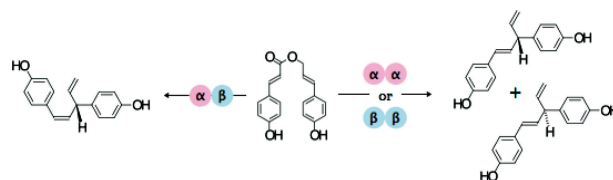


1106

Subunit composition of hinokiresinol synthase controls enantiomeric selectivity in hinokiresinol formation

Masaomi Yamamura, Shiro Suzuki, Takefumi Hattori and Toshiaki Umezawa*

Subunit composition of hinokiresinol synthase can control not only *cis/trans* isomerism but also enantioselectivity in hinokiresinol formation

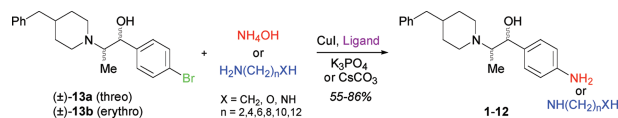


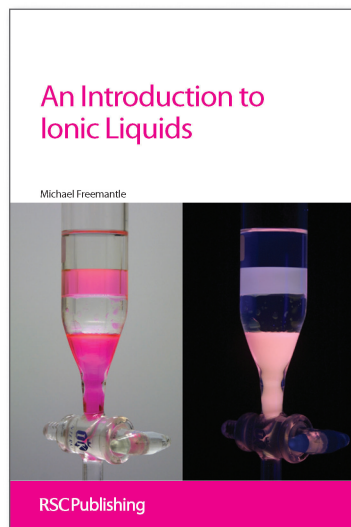
1111

Copper-catalyzed amination of (bromophenyl)ethanolamine for a concise synthesis of aniline-containing analogues of NMDA NR2B antagonist ifenprodil

Cédric Bouteiller, Javier Becerril-Ortega, Patrice Marchand, Olivier Nicole, Louisa Barré, Alain Buisson and Cécile Perrio*

Anilines **1–12** were prepared by copper-catalyzed amination of bromoarenes **13** using CuI and *N,N*-diethylsalicylamide, 2,4-pentadione or 2-acetylcyclohexanone as catalytic systems, and were evaluated as NR2B antagonists.





An Introduction to Ionic Liquids

Michael Freemantle

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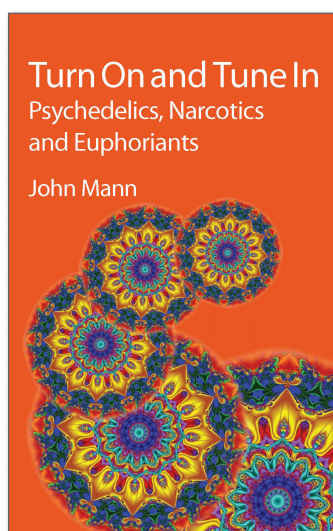
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John Mann

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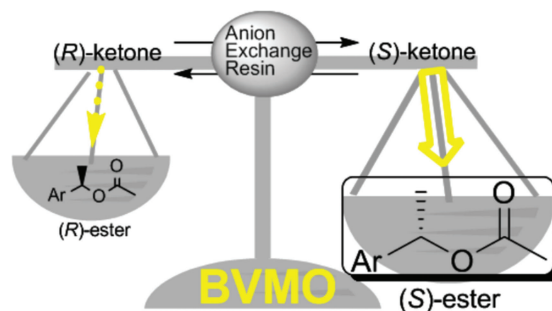
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1121

BVMO-catalysed dynamic kinetic resolution of racemic benzyl ketones in the presence of anion exchange resins

Cristina Rodríguez, Gonzalo de Gonzalo, Ana Rioz-Martínez, Daniel E. Torres Pazmiño, Marco W. Fraaije and Vicente Gotor*

Dynamic kinetic resolutions of different benzyl ketones were performed by combining the selective Baeyer–Villiger oxidation catalysed by HAPMO with anion exchange racemisation in order to obtain the corresponding (*S*)-benzyl esters with high yields and optical purities.

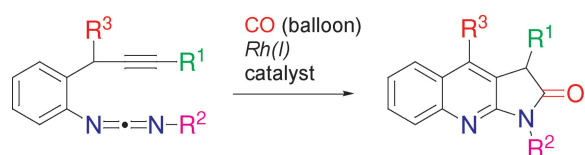


1126

A facile synthesis of pyrrolo[2,3-*b*]quinolines via a Rh(I)-catalyzed carbodiimide–Pauson–Khand-type reaction

Takao Saito,* Naoki Furukawa and Takashi Otani

A Rh(I)-catalyzed Pauson–Khand-type [2 + 2 + 1] cocyclization of *N*-[2-(2-alkyn-1-yl)phenyl]carbodiimides provides a new, straightforward synthetic method for pyrrolo[2,3-*b*]quinolin-2-ones.



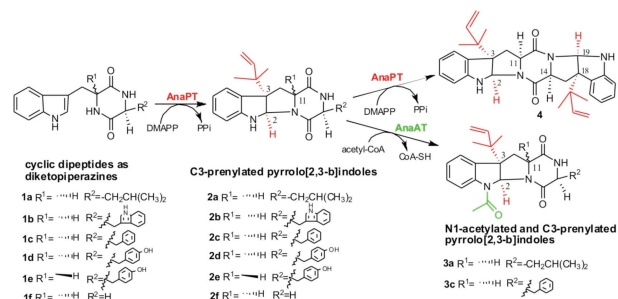
R^1 = Pent, Me, *t*-Bu, Ph, or TBS, R^2 = Pr, Bn, Cy, or Ph, R^3 = H or Me

1133

Reconstruction of pyrrolo[2,3-*b*]indoles carrying an α -configured reverse C3-dimethylallyl moiety by using recombinant enzymes

Wen-Bing Yin, Xiu-Lan Xie, Marco Matuschek and Shu-Ming Li*

Nine reversely C3-prenylated pyrrolo[2,3-*b*]indoles were successfully prepared by using recombinant AnaPT and AnaAT. An α -configured fused ring between the indoline and the diketopiperazine rings was introduced.

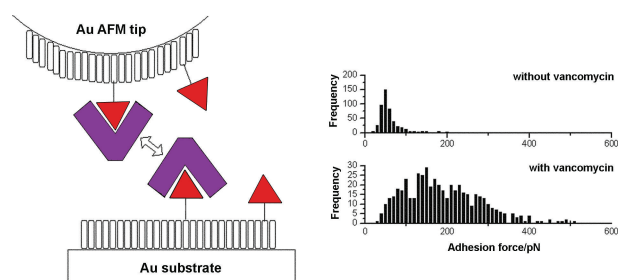


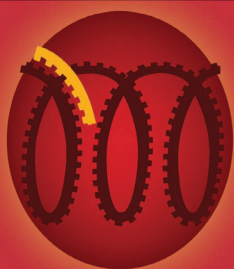
1142

Vancomycin dimer formation between analogues of bacterial peptidoglycan surfaces probed by force spectroscopy

Matthew Batchelor,* Dejian Zhou, Matthew A. Cooper, Chris Abell and Trevor Rayment

Force spectroscopy was used to investigate the rupture of interfacial vancomycin dimer complexes formed between pairs of vancomycin molecules when bound to model bacterial cell-wall surfaces.





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Dmitry Samarsky, VP, Technology Development, RXi Pharmaceuticals

Carl Novina, Assistant Professor, Dana-Farber Cancer Institute & Harvard Medical School

Andrei Thomas-Tikhonenko, Associate Professor, University of Pennsylvania

Carlos Croce, Professor, Ohio State University

Dan Peer, Head, Laboratory of Nanomedicine, Tel Aviv University

Bino John, Assistant Professor, University of Pittsburgh

Paul White, Senior Lecturer, Monash University

Aldo Roccaro, Instructor in Medicine, Harvard Medical School, Dana-Farber Cancer Institute

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PAPERS

1149

A new facile synthesis of 3-amidoindole derivatives and their evaluation as potential GSK-3 β inhibitors

Anahit Pews-Davtyan, Annegret Tillack, Anne-Caroline Schmöle, Stefanie Ortinau, Moritz J. Frech, Arndt Rolfs* and Matthias Beller*

3-Amidoindoles were synthesized from commercially available arylhydrazines and propargylamines over Zn-salt mediated one pot procedure in excellent regioselectivity and up to 94% yield.

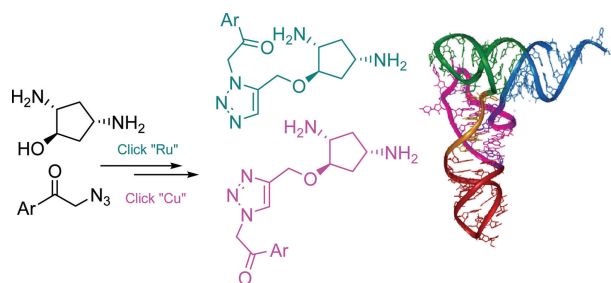


1154

Tether influence on the binding properties of tRNA^{Lys3} ligands designed by a fragment-based approach

Roba Moumné, Valéry Larue, Bili Seijo, Thomas Lecourt, Laurent Micouin* and Carine Tisné*

1,5 triazole derivatives bind to tRNA^{Lys3} with similar affinity but different selectivity than their corresponding 1,4 isomers.

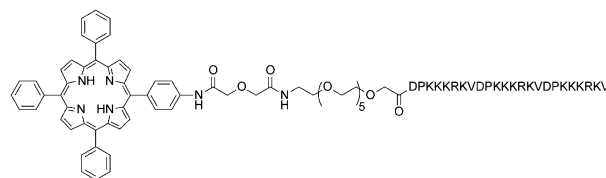


1160

Influence of the number and distribution of NLS peptides on the photosensitizing activity of multimeric porphyrin–NLS

Martha Sibrian-Vazquez, Timothy J. Jensen and M. Graça H. Vicente*

The total synthesis and *in vitro* biological properties of a new series of multimeric porphyrin–NLS conjugates bearing two, three or four peptides with the minimum sequence PKKKRKV are described. The mono- and di-substituted photosensitizers bearing one or two PEG linkers and up to three peptide sequences were found to be the most phototoxic toward human carcinoma HEP2 cells.

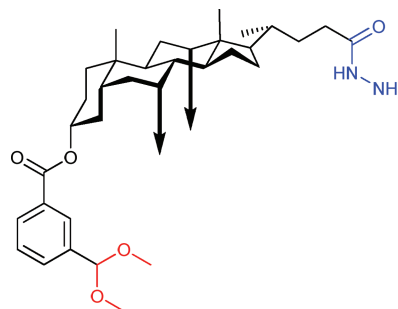


1173

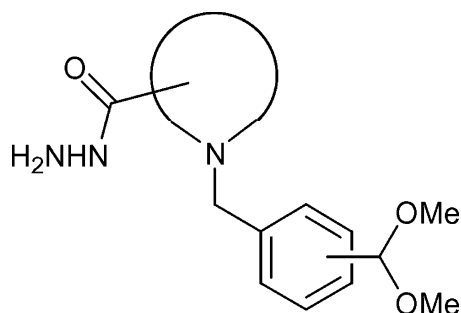
Dynamic combinatorial chemistry with hydrazones: cholate-based building blocks and libraries

Mark G. Simpson, Michael Pittelkow,* Stephen P. Watson and Jeremy K. M. Sanders*

The synthesis and properties of a series of cholate-based building blocks for dynamic combinatorial libraries utilising hydrazone chemistry are described along with a number of exchange experiments demonstrating self-sorting.



1181

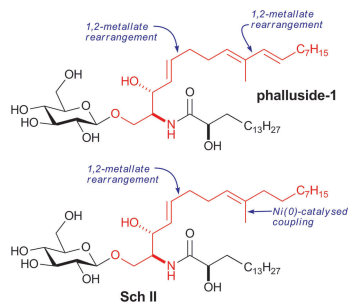


Dynamic combinatorial chemistry with hydrazones: libraries incorporating heterocyclic and steroidal motifs

Mark G. Simpson, Michael Pittelkow,* Stephen P. Watson and Jeremy K. M. Sanders*

The synthesis and properties of a series of heterocycle-based building blocks for dynamic combinatorial libraries utilising hydrazone chemistry is described along with mixing experiments with steroid based hydrazone building blocks.

1188

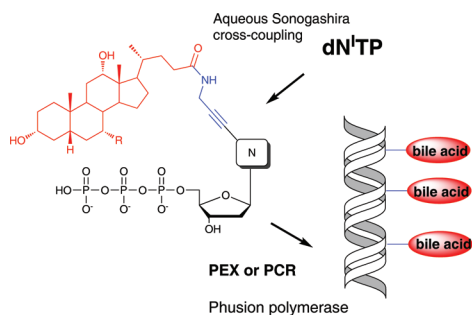


Synthesis of phalluside-1 and Sch II using 1,2-metallate rearrangements

Fiona J. Black and Philip J. Kocienski*

For the first time a synthesis of (4*E*,8*E*,10*E*)-9-methyl-4,8,10-sphingatrienine, the acid-labile core component of marine sphingolipids, has been achieved using a fragment linkage strategy based on copper-mediated 1,2-metallate rearrangements. A related synthesis of Sch II was also accomplished.

1194

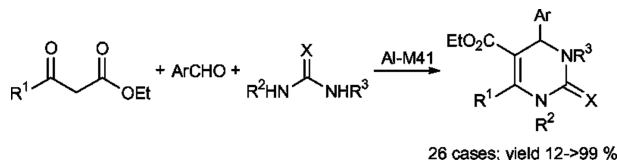


Synthesis of nucleoside and nucleotide conjugates of bile acids, and polymerase construction of bile acid-functionalized DNA

Satu Ikonen, Hana Macíčková-Cahová, Radek Pohl, Miloslav Šanda and Michal Hocek*

Sonogashira cross-couplings of halogenated nucleosides and nucleoside triphosphates with bile-acid acetylenes gave steroid–nucleos(t)ide conjugates that were incorporated to DNA by polymerase.

1202



Synthesis of Biginelli dihydropyrimidinone derivatives with various substituents on aluminium-planted mesoporous silica catalyst

Hiroaki Murata, Haruro Ishitani and Masakazu Iwamoto*

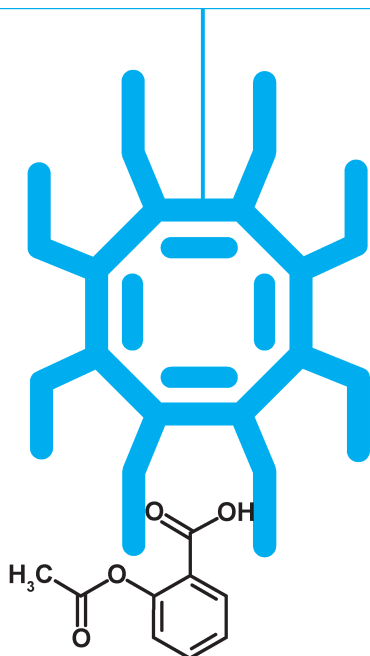
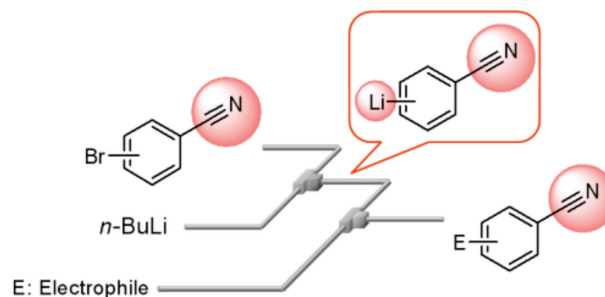
Al-planted mesoporous silica with Si/Al ratios of 45–35 catalyzed the title reaction with good to excellent yields; some of the products have been very difficult to synthesize until now.

1212

Generation and reaction of cyano-substituted aryllithium compounds using microreactors

Aiichiro Nagaki, Heejin Kim, Hirotsugu Usutani,
Chika Matsuo and Jun-ichi Yoshida*

An effective method for the generation and reaction of aryllithium compounds bearing a cyano group has been developed using microflow systems.



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