

Organic & Biomolecular Chemistry

www.rsc.org/obc



ISSN 1477-0520



COMMUNICATION

Sofia I. Pascu, Yun-Bao Jiang, Tony D. James *et al.*
Synthesis and evaluation of a boronate-tagged 1,8-naphthalimide probe for fluoride recognition

Organic & Biomolecular Chemistry

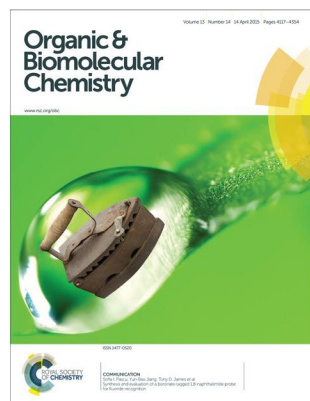
An international journal of synthetic, physical and biomolecular organic chemistry

www.rsc.org/obc

The Royal Society of Chemistry is the world's leading chemistry community. Through our high impact journals and publications we connect the world with the chemical sciences and invest the profits back into the chemistry community.

IN THIS ISSUE

ISSN 1477-0520 CODEN OBCRAK 13(14) 4117–4354 (2015)

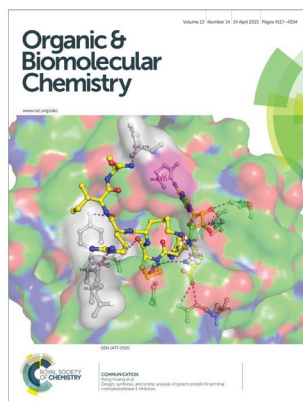


Cover

See Sofia I. Pascu,
Yun-Bao Jiang,
Tony D. James *et al.*,
pp. 4143–4148.

© Mackon and Dreamstime.
com are acknowledged for
the water droplet photo.
© Fastof and Dreamstime.
com are acknowledged for
the old iron photo.

Image reproduced by
permission of Tony D. James
from *Org. Biomol. Chem.*,
2015, **13**, 4143.



Inside cover

See Rong Huang *et al.*,
pp. 4149–4154.

Image reproduced by
permission of Rong Huang
from *Org. Biomol. Chem.*,
2015, **13**, 4149.

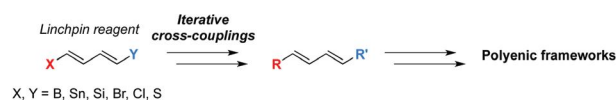
REVIEW

4129

Linchpin dienes: key building-blocks in the synthesis of polyenic frameworks

J. Cornil, A. Guérinot and J. Cossy*

This review focuses on the preparation of dienic linchpin reagents and on their use in the synthesis of polyenic frameworks.



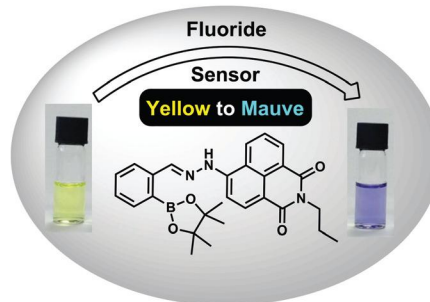
COMMUNICATIONS

4143

Synthesis and evaluation of a boronate-tagged 1,8-naphthalimide probe for fluoride recognition

Su-Ying Xu, Xiaolong Sun, Haobo Ge,
Rory L. Arrowsmith, John S. Fossey, Sofia I. Pascu,*
Yun-Bao Jiang* and Tony D. James*

A biocompatible fluoride receptor has been developed where the interaction between the boronic acid ester and amine (NH) results in fluoride ion selectivity and enhanced fluorescence quenching.



Editorial staff

Editor

Richard Kelly

Deputy editor

Marie Cote

Editorial production manager

Helen Saxton

Development editor

James Anson

Publishing editors

Nicola Burton, Zoe Karthäuser, Elisa Meschini, Roxane Owen, Simon Rankmore, Donna Smith

Publishing assistants

Emily Finney, Rosalind Searle

Publisher

Emma Wilson

For queries about submitted papers, please contact Helen Saxton, Editorial production manager in the first instance. E-mail: obc@rsc.org

For pre-submission queries please contact Richard Kelly, Editor. Email: obc-rsc@rsc.org

Organic & Biomolecular Chemistry (print: ISSN 1477-0520; electronic: ISSN 1477-0539) is published 48 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF.

All orders, with cheques made payable to the Royal Society of Chemistry, should be sent to RSC Order Department, Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, CB4 0WF, UK. Tel +44 (0)1223 432398; E-mail orders@rsc.org

2015 Annual (print+electronic) subscription price: £4572; US\$8534. 2015 Annual (electronic) subscription price: £4343; US\$8107. Customers in Canada will be subject to a surcharge to cover GST. Customers in the EU subscribing to the electronic version only will be charged VAT. If you take an institutional subscription to any RSC journal you are entitled to free, site-wide web access to that journal. You can arrange access via Internet Protocol (IP) address at www.rsc.org/ip. Customers should make payments by cheque in sterling payable on a UK clearing bank or in US dollars payable on a US clearing bank.

The Royal Society of Chemistry takes reasonable care in the preparation of this publication but does not accept liability for the consequences of any errors or omissions. Inclusion of an item in this publication does not imply endorsement by The Royal Society of Chemistry of the content of the original documents to which that item refers.

Advertisement sales: Tel +44 (0) 1223 432246; Fax +44 (0) 1223 426017; E-mail advertising@rsc.org

For marketing opportunities relating to this journal, contact marketing@rsc.org



Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

www.rsc.org/obc

Organic & Biomolecular Chemistry brings together molecular design, synthesis, structure, function and reactivity in one journal. Broad in scope, it publishes research and reviews on topics across organic synthesis, physical organic chemistry, supramolecular chemistry and chemical biology.

Editorial board

Chair

Andrei Yudin, University of Toronto, Canada

Associate editors

Margaret Brimble, University of Auckland, New Zealand
Jin-Guan Yu, Scripps Research Institute, La Jolla, CA, USA

Editorial board members

Ashraf Brik, Technion-Israel Institute of Technology, Israel
Pauline Chiu, University of Hong Kong, China
Jonathan Clayden, University of Manchester, UK
Anthony Davis, University of Bristol, UK

Christian Hertweck, Leibniz-Institute Jena, Germany
Kenichiro Itami, Nagoya University, Japan
Dean Tantillo, UC Davis, USA
Qi-Lin Zhou, Nankai University, China

Advisory board

Kyo Han Ahn, Pohang University of Science and Technology, Korea
Fredrik Almqvist, Umeå University, Sweden

Jeffrey Bode, ETH Zurich, Switzerland
Barry Carpenter, Cardiff University, UK

David Chen, Seoul National University, Korea

Shunsuke Chiba, Nanyang Technological University, Singapore

Sheng-Hsien Chiu, National Taiwan University, Chinese Taipei

Luiz Carlos Dias, State University of Campinas, Brazil

Antonio Echavarren, Autonomous University of Madrid, Spain

Jonathan Ellman, Yale University, USA
Margaret Faul, Amgen, USA

Ben Feringa, University of Groningen, The Netherlands

Amar Flood, Indiana University Bloomington, USA

Nobutaka Fujii, Kyoto University, Japan

Carmen Galan, University of Bristol, UK

Sam Gellman, University of Wisconsin, USA

Christian Hackenberger, Free University Berlin, Germany

Mimi Hii, Imperial College London, UK

Krishna Kaliappan, IITB, India

Steven V. Ley, University of Cambridge, UK

Shih-Yuan Liu, University of Oregon, USA

Stephen Loeb, University of Windsor, Canada

David Lupton, Monash University, Australia

Ilan Marek, Israel Institute of Technology, Israel

Keiji Maruoka, Kyoto University, Japan

Cristina Nevado, University of Zürich, Switzerland

Dhevalapally B. Ramachary, University of Hyderabad, India

Viresh Rawal, University of Chicago, USA

Mark Rizzacasa, University of Melbourne, Australia

Richmond Sarpong, University of California, Berkeley, USA

Paolo Scrimin, University of Padua, Italy

Oliver Seitz, Humboldt University of Berlin, Germany

Jay Siegel, University of Zürich, Switzerland

Tibor Soos, Hungarian Academy of Sciences, Hungary

Corey Stephenson, University of Michigan, USA

Mark Taylor, University of Toronto, Canada

Dirk Trauner, Ludwig-Maximilian University Munich, Germany

Bruce Turnbull, University of Leeds, UK

Georgios Vassilikogiannakis, University of Crete, Greece

Helma Wennemers, University of Basel, Switzerland

Peter Wipf, University of Pittsburgh, USA

Shuli You, Shanghai Institute of Organic Chemistry, China

Li He Zhang, Peking University, China

Jian Zhou, East China Normal University, China

Information for authors

Full details on how to submit material for publication in *Organic & Biomolecular Chemistry* are given in the Instructions for Authors (available from <http://www.rsc.org/authors>).

Submissions should be made via the journal's homepage: <http://www.rsc.org/obc>.

Authors may reproduce/republish portions of their published contribution without seeking permission from the RSC, provided that any such republication is accompanied by an acknowledgement in the form: (Original Citation)—Reproduced by permission of The Royal Society of Chemistry.

This journal is ©The Royal Society of Chemistry 2015. Apart from fair dealing for the purposes of research or private study for non-commercial purposes, or criticism or review, as permitted under the Copyright, Designs and

Patents Act 1988 and the Copyright and Related Rights Regulation 2003, this publication may only be reproduced, stored or transmitted, in any form or by any means, with the prior permission in writing of the Publishers or in the case of reprographic reproduction in accordance with the terms of licences issued by the Copyright Licensing Agency in the UK. US copyright law is applicable to users in the USA.

The Royal Society of Chemistry takes reasonable care in the preparation of this publication but does not accept liability for the consequences of any errors or omissions.

© The paper used in this publication meets the requirements of ANSI/NISO Z39.48-1992 (Permanence of Paper).

Registered Charity No. 207890.

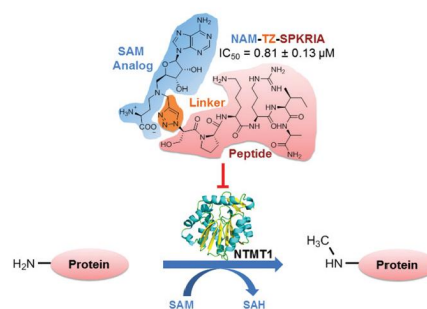
COMMUNICATIONS

4149

Design, synthesis, and kinetic analysis of potent protein N-terminal methyltransferase 1 inhibitors

Gang Zhang, Stacie Lynn Richardson, Yunfei Mao and Rong Huang*

A novel clicked bisubstrate analogue (NAM-TZ-SPKRIA) was designed and synthesized to be a potent, selective, and first NTMT1 inhibitor.

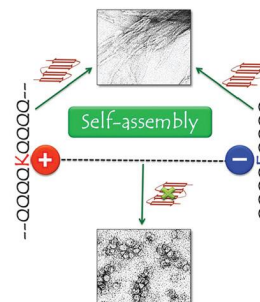


4155

Amyloid nanospheres from polyglutamine rich peptides: assemblage through an intermolecular salt bridge interaction

Rahul Mishra and Ashwani K. Thakur*

Amyloid fiber formation by two polyglutamine peptides through a nucleation polymerization pathway. An intermolecular salt bridge between the positively charged lysine and the negatively charged glutamate induces the formation of nanospherical amyloids through a non-nucleated pathway.

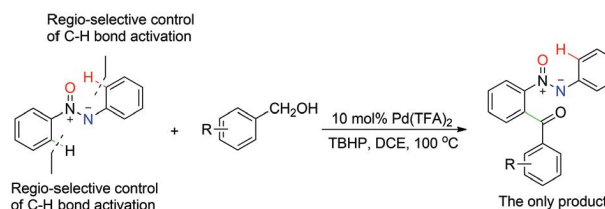


4160

Palladium-catalyzed regio-selective oxidative C–H bond acylation of azoxybenzenes with alcohols

Lekai Hou, Xiangxiang Chen, Shuang Li, Suxian Cai, Yanxia Zhao, Meng Sun* and Xiao-Juan Yang

A novel catalytic system for the *ortho*-acetyl functionalization of azoxybenzenes *via* a regio-selective directing-group-assisted strategy was developed.

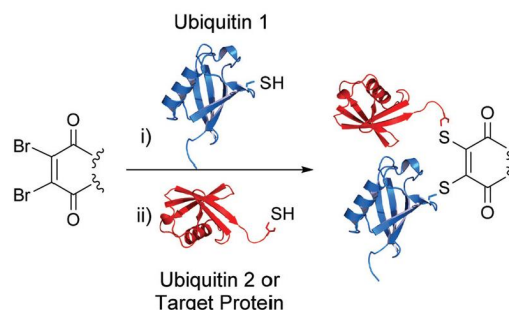


4165

A novel synthetic chemistry approach to linkage-specific ubiquitin conjugation

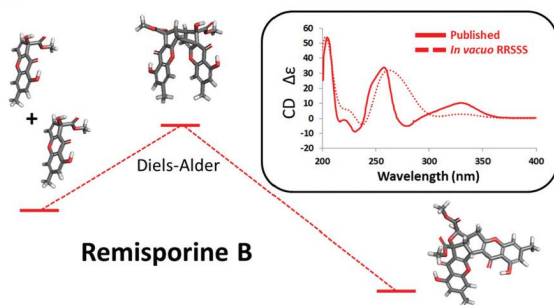
Rachel E. Morgan, Vijay Chudasama, Paul Moody, Mark E. B. Smith and Stephen Caddick*

Site-specific ubiquitin cysteine mutants enable an elegant method for the linkage-specific conjugation of ubiquitins through dibromomaleimides and dibromopyridazinediones.



COMMUNICATIONS

4169



Absolute configuration of remisporines A & B

Edward C. Sherer,* James R. Cheeseman and R. Thomas Williamson

The absolute configuration of remisporine B was determined based on a comparison of experimental and calculated electronic circular dichroism (ECD) spectra.

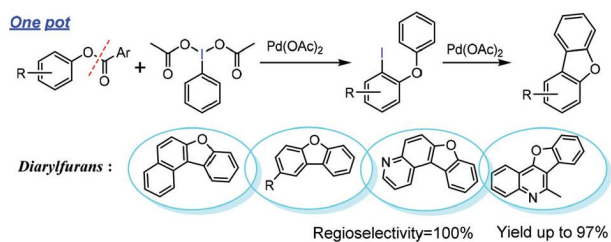
4174

Copper-catalyzed asymmetric allylation of chiral *N*-tert-butanesulfinyl imines: dual stereocontrol with nearly perfect diastereoselectivity

Yi-Shuang Zhao, Qiang Liu, Ping Tian,* Jing-Chao Tao* and Guo-Qiang Lin

Copper-catalyzed asymmetric allylation of chiral *N*-tert-butanesulfinyl imines has been described.

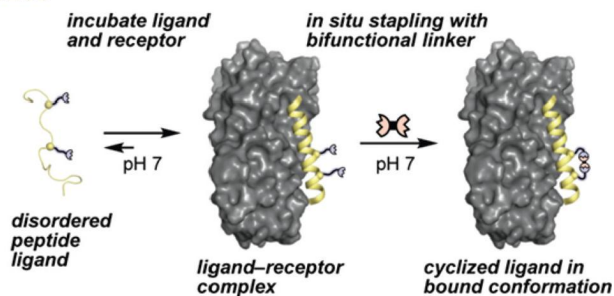
4179

One pot synthesis of diarylfurans from aryl esters and PhI(OAc)₂ via palladium-associated iodonium ylides

Yong-Sheng Bao,* Bao Agula, Bao Zhaorigetu, Meilin Jia and Menghe Baiyin

Palladium-catalyzed intermolecular cyclization for the synthesis of various diarylfurans from aryl esters and PhI(OAc)₂ via iodonium ylide rearrangement induced by Pd(II) has been reported.

4183



Receptor-templated stapling of intrinsically disordered peptide ligands

Conor M. Haney and W. Seth Horne*

We report here a method for peptide stapling where a protein receptor guides the reaction by acting as a template that folds a disordered ligand into a bioactive state prior to cyclization.

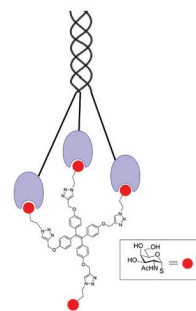
PAPERS

4190

Bi- to tetravalent glycoclusters presenting GlcNAc/GalNAc as inhibitors: from plant agglutinins to human macrophage galactose-type lectin (CD301) and galectins

Sabine André, Shane O'Sullivan, Christiane Koller, Paul V. Murphy* and Hans-Joachim Gabius

The trimeric C-type lectin MGL is involved in activating immune defence and virus uptake, thus becoming a target for inhibitor design. A tetravalent cluster with α -S-GalNAc proved to be effective in the nM range.

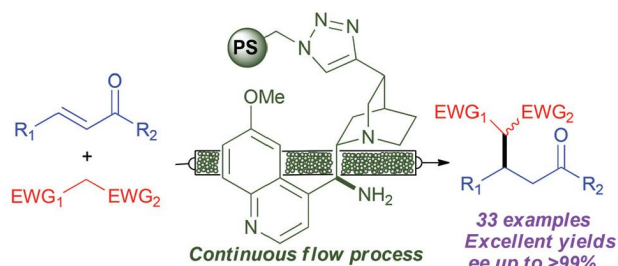


4204

A polystyrene-supported 9-amino(9-deoxy)epi quinine derivative for continuous flow asymmetric Michael reactions

Javier Izquierdo, Carles Ayats, Andrea H. Henseler and Miquel A. Pericàs*

A polystyrene (PS)-supported 9-amino(9-deoxy)epi quinine derivative catalyzes Michael reactions affording excellent levels of conversion and enantioselectivity using different nucleophiles and structurally diverse enones under continuous flow conditions.



4210

Strategically designed biomodel: engineering C3–C4 cleavage of D-fructose

Palwinder Singh,* Arun Kumar, Sukhmeet Kaur and Amrinder Singh

Amongst a library of aldolase inspired, rationally designed compounds, the acridine derivative carrying a (S)-Tyr-Gly-(S)-Lys tripeptide selectively effected C3–C4 scissoring of D-fructose and produced D-glyceraldehyde and dihydroxyacetone.

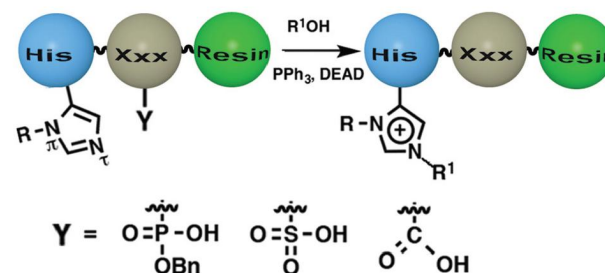


4221

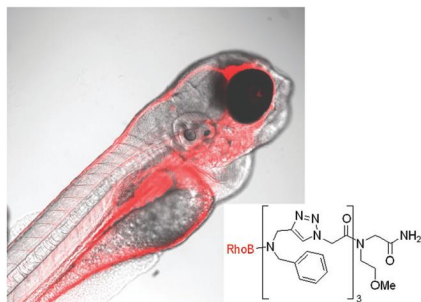
Mitsunobu mischief: neighbor-directed histidine N(τ)-alkylation provides access to peptides containing selectively functionalized imidazolium heterocycles

Wen-Jian Qian and Terrence R. Burke, Jr.*

Selective on-resin histidine N(τ)-alkylation under Mitsunobu conditions is achieved by the coordinated participation of a proximal acidic residue.



4226

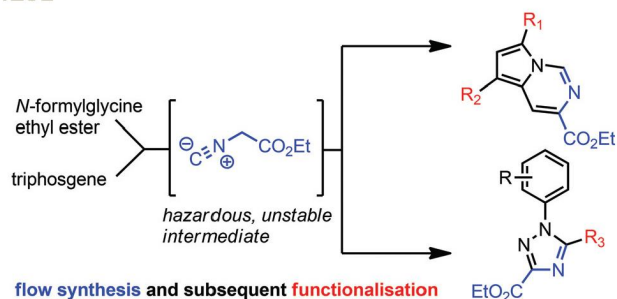


Functionalized triazolo-peptoids – a novel class for mitochondrial targeted delivery

Daniela Althun, Franziska Röncke, Daniel Fürniss, Jasmin Guan, Isabelle Wellhöfer, Nicole Jung, Ute Schepers* and Stefan Bräse*

Here we introduce linear 1,4-triazolo-peptoids as a novel class of cell penetrating peptidomimetics suitable as organ targeting molecular transporters of bioactive cargo.

4231

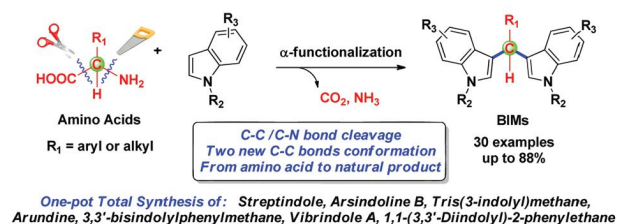


Flow synthesis of ethyl isocyanoacetate enabling the telescoped synthesis of 1,2,4-triazoles and pyrrolo-[1,2-c]pyrimidines

Marcus Baumann, Antonio M. Rodriguez Garcia and Ian R. Baxendale*

The efficient flow synthesis of important heterocyclic building blocks based on the 1,2,4-triazole and pyrrolo[1,2-c]pyrimidine scaffold has been achieved.

4240

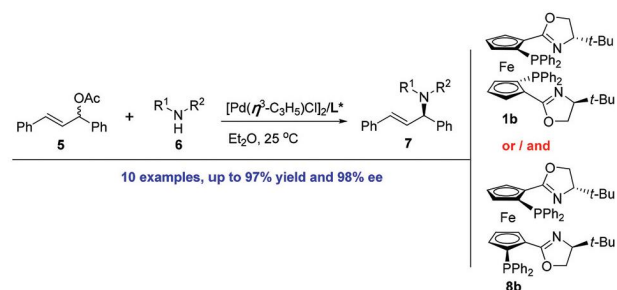


One-pot total synthesis of streptindole, arsindoline B and their congeners through tandem decarboxylative deaminative dual-coupling reaction of amino acids with indoles

Jiachen Xiang, Jungang Wang, Miao Wang, Xianggao Meng and Anxin Wu*

This paper described a decarboxylative deaminative dual-coupling reaction of amino acids with indoles to afford BIM scaffolds and its further application to the one-pot total synthesis of natural products.

4248



Pd-catalyzed asymmetric allylic amination using easily accessible metallocenyl P,N-ligands

Hongwei Wu, Fang Xie, Yanlan Wang, Xiaohu Zhao, Delong Liu* and Wanbin Zhang*

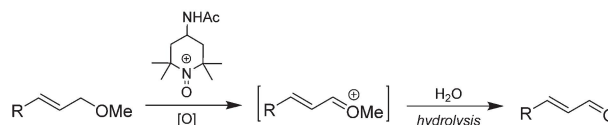
Pd-catalyzed asymmetric allylic aminations were carried out efficiently using both C_2 -symmetric and non- C_2 -symmetric metallocenyl P,N-ligands. A more accessible mixed ligand system of the above two was then examined, providing the amination product with high yield and excellent enantioselectivity.

4255

Oxidative cleavage of allyl ethers by an oxoammonium salt

Christopher B. Kelly, John M. Ovian, Robin M. Cywar, Taylor R. Gosselin, Rebecca J. Wiles and Nicholas E. Leadbeater*

A method to oxidatively cleave allyl ethers to their corresponding aldehydes mediated by an oxoammonium salt is described. Using a biphasic solvent system and mild heating, cleavage proceeds readily, furnishing a variety of α,β -unsaturated aldehydes and ketones.

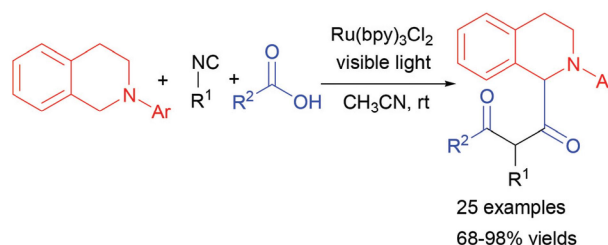


4260

Visible light mediated sp^3 C–H bond functionalization of *N*-aryl-1,2,3,4-tetrahydroisoquinolines via Ugi-type three-component reaction

Yunyun Chen and Gaofeng Feng*

An efficient and high yield process for sp^3 C–H bond functionalization of *N*-aryl-1,2,3,4-tetrahydroisoquinolines is disclosed.

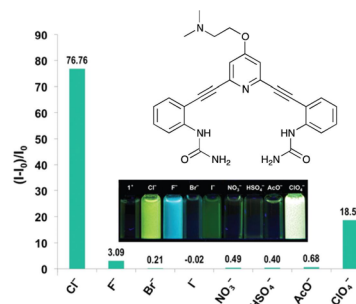


4266

"Off-on" aggregation-based fluorescent sensor for the detection of chloride in water

Michelle M. Watt, Jeffrey M. Engle, Kurtis C. Fairley, Timothy E. Robitshek, Michael M. Haley* and Darren W. Johnson*

A new class of 2,6-bis(2-anilinoethynyl)pyridine bisureas exhibits selective turn-on fluorescence for chloride in water.

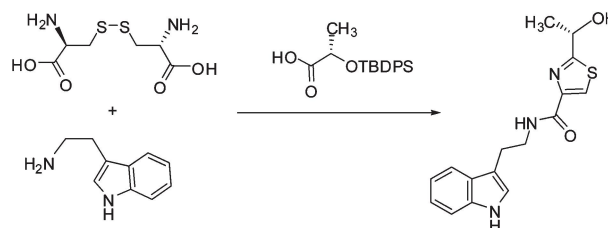


4271

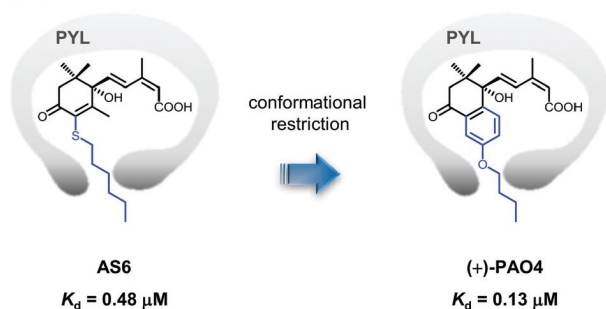
Synthesis and structural reconfirmation of bacillamide B

Xue Sun, Yi Liu, Jun Liu, Guofeng Gu and Yuguo Du*

Synthesis of bacillamide B was accomplished, the absolute configuration was reconfirmed as *S*, and the specific optical rotation was revised to (–).



4278



Conformationally restricted 3'-modified ABA analogs for controlling ABA receptors

Jun Takeuchi, Toshiyuki Ohnishi, Masanori Okamoto and Yasushi Todoroki*

(+)-PAO4 is a conformationally restricted analog of AS6 that was synthesized to improve the affinity for PYL proteins.

4289

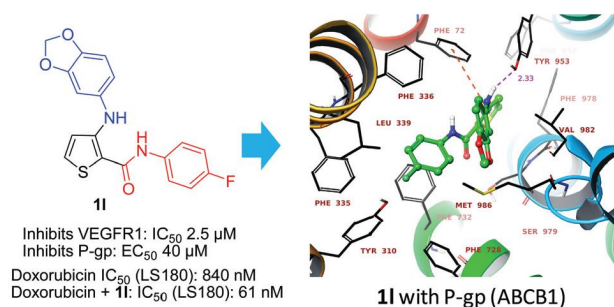


Benzimidazoles and benzoxazoles via the nucleophilic addition of anilines to nitroalkanes

Alexander V. Aksenov,* Alexander N. Smirnov, Nicolai A. Aksenov, Asiyat S. Bijieva, Inna V. Aksenova and Michael Rubin*

PPA-induced umpolung triggers efficient nucleophilic addition of unactivated anilines to nitroalkanes to afford benzoxazoles and benzimidazoles.

4296

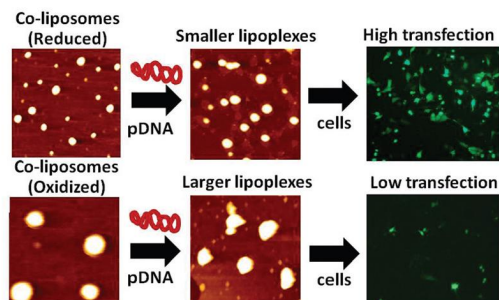


3-(Benzo[d][1,3]dioxol-5-ylamino)-N-(4-fluorophenyl)thiophene-2-carboxamide overcomes cancer chemoresistance via inhibition of angiogenesis and P-glycoprotein efflux pump activity

Ramesh Mudududdla, Santosh K. Guru, Abubakar Wani, Sadhana Sharma, Prashant Joshi, Ram A. Vishwakarma, Ajay Kumar,* Shashi Bhushan* and Sandip B. Bharate*

Thiophene-2-carboxamides displayed dual inhibition of angiogenesis and P-gp efflux pumps.

4310



Efficacious redox-responsive gene delivery in serum by ferrocenylated monomeric and dimeric cationic cholesterols

Gururaja Vulugundam, Krishan Kumar, Paturu Kondaiah and Santanu Bhattacharya*

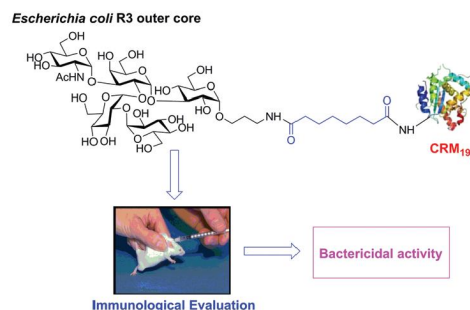
New redox-active monomeric and dimeric ferrocenylated cationic cholesterols for gene transfection.

4321

Chemical synthesis of the outer core oligosaccharide of *Escherichia coli* R3 and immunological evaluation

Wenjing Shang, Zhongying Xiao, Zaikuan Yu, Na Wei, Guohui Zhao, Qing Zhang, Mohui Wei, Xuan Wang, Peng George Wang* and Tiejai Li*

An all α -linked *Escherichia coli* R3 outer core pentasaccharide was first synthesized. Its corresponding glycoconjugate can elicit specific anti-pentasaccharide antibodies with *in vitro* bactericidal activity.

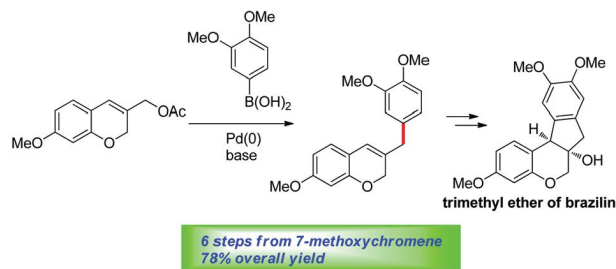


4331

A concise synthetic approach to brazilin via Pd-catalyzed allylic arylation

Youngeun Jung and Ikyon Kim*

A short synthetic route to the trimethyl ether of brazilin was developed in 6 steps from 7-methoxychromene with 78% overall yield.

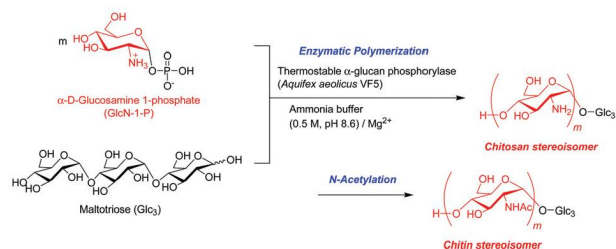


4336

Synthesis of chitin and chitosan stereoisomers by thermostable α -glucan phosphorylase-catalyzed enzymatic polymerization of α -D-glucosamine 1-phosphate

Jun-ichi Kadokawa,* Riko Shimohigoshi, Kento Yamashita and Kazuya Yamamoto

Chitosan and chitin stereoisomers were successfully synthesized by thermostable α -glucan phosphorylase-catalyzed enzymatic polymerization of α -D-glucosamine 1-phosphate and subsequent N-acetylation.

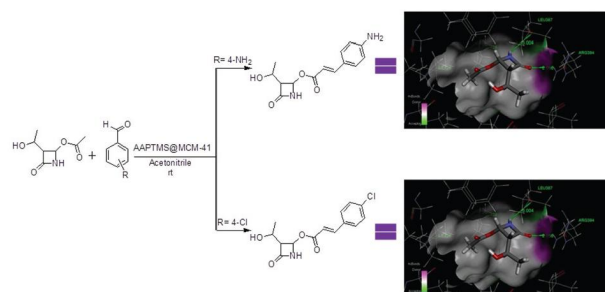


4344

Novel carbapenem chalcone derivatives: synthesis, cytotoxicity and molecular docking studies

Devendar Reddy Kommidi,* Ramakanth Pagadala, Surjyakanta Rana, Parvesh Singh, Suhas A. Shintre, N. A. Koorbanally, Sreekantha B. Jonnalagadda and Brenda Moodley*

One-pot efficient synthetic protocol is described for the synthesis of carbapenem chalcone derivatives using AAPTMS@MCM-41 heterogeneous catalyst.



CORRECTION

4351

Correction: A novel Prins cascade process for the stereoselective synthesis of oxa-bicycles

B. V. Subba Reddy,* A. Venkateswarlu, B. Sridevi, Kanakaraju Marumudi, A. C. Kunwar and G. Gayatri