

Organic & Biomolecular Chemistry

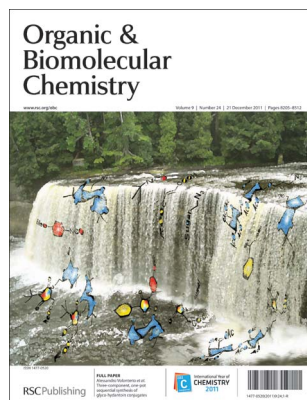
An international journal of synthetic, physical and biomolecular organic chemistry

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IN THIS ISSUE

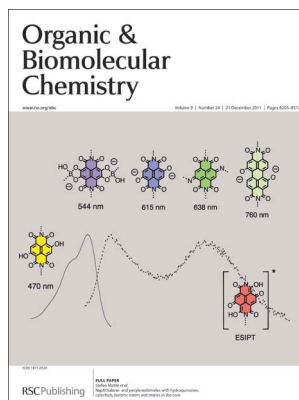
ISSN 1477-0520 CODEN OBCRAK 9(24) 8205–8512 (2011)



Cover

See Alessandro Volonterio *et al.*, pp. 8379–8392.

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Inside cover

See Stefan Matile *et al.*, pp. 8246–8252.

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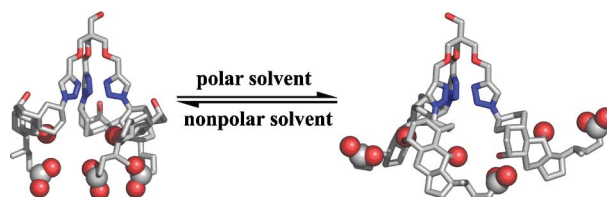
COMMUNICATIONS

8220

Construction of a tunable metallohydrolase center on an invertible molecular pocket

Zhiming Zhang, Junqiu Liu,* Quan Luo, Jiawei Zhang, Jiayun Xu and X. X. Zhu*

An artificial metallohydrolase with an invertible amphiphilic cavity can efficiently modulate the catalytic activity by changing the polarity of the solvent media.

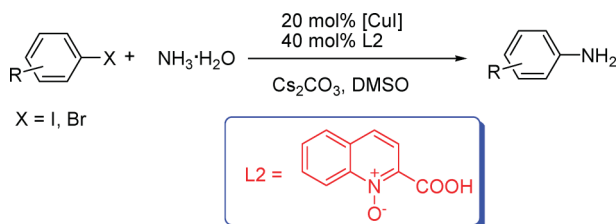


8224

An efficient copper-catalyzed synthesis of anilines by employing aqueous ammonia

Xin Zeng, Wenming Huang, Yatao Qiu and Sheng Jiang*

Under the catalysis of CuI/2-carboxylic acid-quinoline-*N*-oxide, the cross coupling reactions between aryl iodides or bromides and aqueous ammonia proceed very well to afford *N*-unprotected aniline derivatives in excellent yields. This inexpensive catalytic system shows great functional group tolerance and excellent reaction selectivity.



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Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

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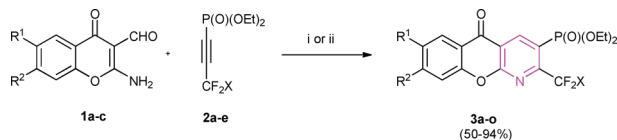
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COMMUNICATIONS

8228

CF₂-Containing acetylenephosphonates in heterocyclization reactions: the first synthesis of 2-difluoromethyl azaxanth-3-ylphosphonates

Blazej Duda, Sergey N. Tverdomed* and Gerd-Volker Röschenthaler*



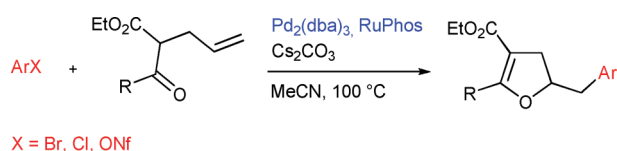
i: *i*-Pr₂NEt, DMSO, RT, 4-12h (X = F, Cl, H, CF₃); ii: K₂CO₃ (5mol%), DMF, RT, 8-9h (X = Br)
R^{1,2} = H, Cl, CH₃

Acetylenephosphonates carrying the XCF₂ group have been studied in a base-mediated heterocyclization reaction with selected 2-amino-3-formylchromones to give 2-difluoromethyl azaxanth-3-ylphosphonates. The presence of the fluorinated substituent determined the regioselectivity as well as the reactivity of this process.

8233

Functionalized 2,3-dihydrofurans via palladium-catalyzed oxyarylation of α -allyl- β -ketoesters

Sandro Cacchi,* Giancarlo Fabrizi, Antonella Goggiamani, Antonia Iazzetti, David Madec, Giovanni Poli and Guillaume Prestat



A palladium-catalyzed synthesis of functionalized 2,3-dihydrofurans from α -allyl- β -ketoesters and (hetero)aryl bromides, chlorides, and nonaflates.

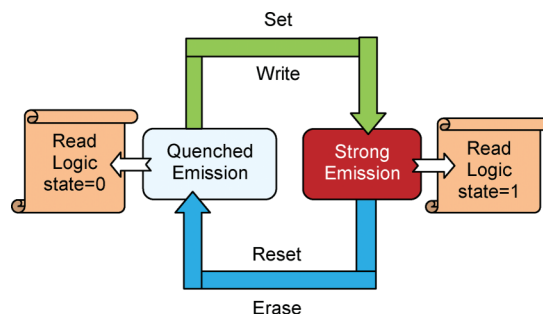
PAPERS

8237

Thiacalix[4]arene based reconfigurable molecular switches: set-reset memorized sequential device

Manoj Kumar,* Rajesh Kumar and Vandana Bhalla

Naphthyl-appended chemosensors based on thiacalix[4]arenes of *cone* and 1,3-*alternate* conformations have been synthesized and evaluated for their fluorescence behavior toward different cations and anions, which mimics a "set-reset" molecular level device.

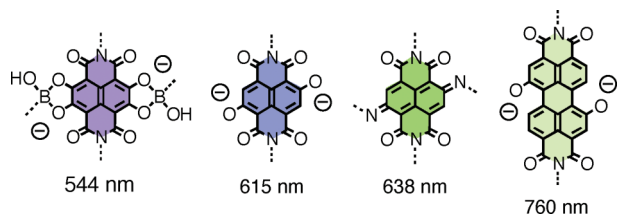


8246

Naphthalene- and perylene diimides with hydroquinones, catechols, boronic esters and imines in the core

Andrea Fin, Irina Petkova, David Alonso Doval, Naomi Sakai, Eric Vauthey and Stefan Matile*

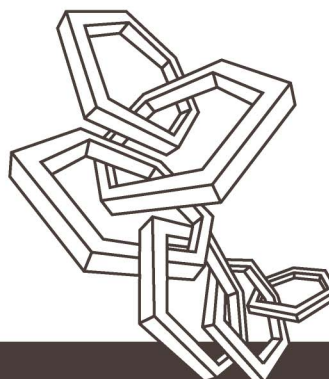
New core substituents for naphthalenediimide and perylene diimide probes are explored, focusing on pH-dependent panchromism, ESIPT emission and oxidative imination.



BOSS | XIII

13th Belgian Organic Synthesis Symposium

July 15>20, 2012, K.U.Leuven, Belgium



Symposium Programme:

- **One-day course** by the recipient of the **Tetrahedron Chair in Organic Synthesis**
 Prof. Ben FERINGA (University of Groningen, The Netherlands)
- **16 plenary lectures**
 Prof. Matthias BELLER (University of Rostock, Germany)
 Prof. Dale L. BOGER (The Scripps Research Institute, USA)
 Prof. Jan-Erling BÄCKVALL (Stockholm University, Sweden)
 Prof. Karl GADEMANN (University of Basel, Switzerland)
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 Prof. István MARKÓ (Université catholique de Louvain, Belgium)
 Prof. Gary A. MOLANDER (Penn Chemistry, University of Pennsylvania, USA)
 Prof. Klaus MÜLLER (F. Hoffmann-la Roche Ltd, Switzerland)
 Prof. Tobias RITTER (Harvard University, USA)
 Prof. Scott SNYDER (Columbia University, USA)
 Prof. Paul A. WENDER (Stanford University, USA)
 Prof. Ming Hua XU (Shanghai Institute of Materia Medica, China)
- **Lecture** delivered by the recipient of the Janssen Pharmaceutica Prize for Creativity in Organic Synthesis
- **Poster sessions**
- **Exhibition**
- **Social activities**

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Janssen Pharmaceutica Prize for Creativity in Organic Synthesis

► Call for nominations

The Janssen Pharmaceutica Prize for Creativity in Organic Synthesis is awarded on a biannual basis, on the occasion of the BOSS, to a chemist under the age of 50 who has made a significant contribution to the field of organic synthesis in the broadest sense. The Prize consists of a trophy, a citation and € 20.000. The laureate of the Prize is expected to deliver a lecture at the BOSS XIII.

All details for the submission of a nomination are available via the symposium website:

www.BOSS13.org

► Deadline for nominations

December 31, 2011

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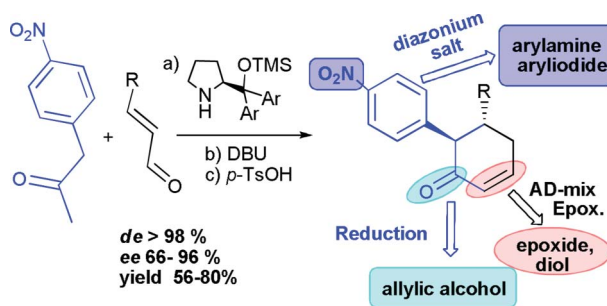


8253

An organocatalytic approach to enantiomerically enriched α -arylcyclohexenones and cyclohexanones

Sara Duce, María Jorge, Inés Alonso,
José Luis García Ruano and M. Belén Cid*

The *p*-nitrophenyl group converts acetone into a versatile nucleophile that affords enantioenriched α -arylcyclohexenones *via* a Michael/aldol reaction/dehydration sequence.

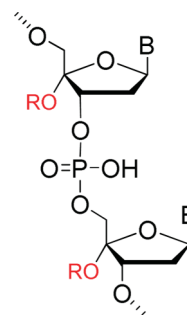


8261

4'-Alkoxy oligodeoxynucleotides: a novel class of RNA mimics

Radek Liboska, Jan Snášel, Ivan Barvík, Miloš Buděšínský,
Radek Pohl, Zdeněk Točík, Ondřej Páv, Dominik Rejman,
Pavel Novák and Ivan Rosenberg*

Solid phase synthesis of 4'-alkoxy-oligodeoxynucleotides and evaluation of their hybridisation properties with DNA and RNA are presented.

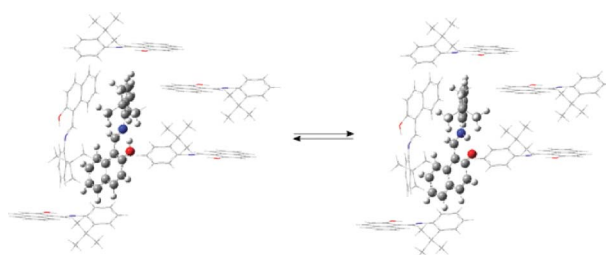


8268

Tautomerism in Schiff bases. The cases of 2-hydroxy-1-naphthaldehyde and 1-hydroxy-2-naphthaldehyde investigated in solution and the solid state

R. Fernando Martínez,* Martín Ávalos, Reyes Babiano,
Pedro Cintas, José L. Jiménez, Mark E. Light and
Juan C. Palacios

A simulation of the crystal lattice unveils the origin of tautomer preferences in Schiff bases derived from hydroxy naphthaldehydes.

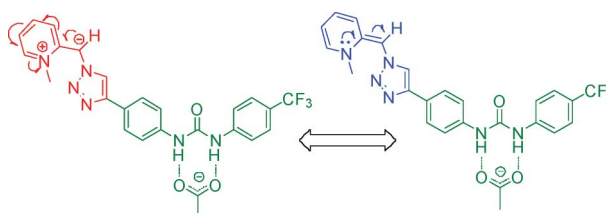


8276

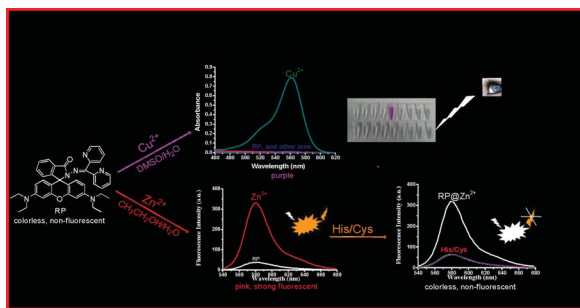
Pyridinium/urea-based anion receptor: methine formation in the presence of basic anions

Greta Bergamaschi, Massimo Boiocchi, Enrico Monzani
and Valeria Amendola*

A new receptor capable of monitoring both bound anion and excess anion in solution, through the development of coloured methine species.



8284

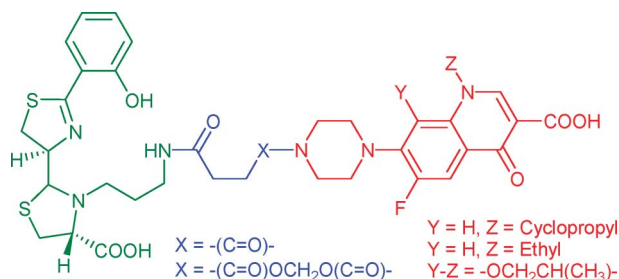


Versatile trifunctional chemosensor of rhodamine derivative for Zn^{2+} , Cu^{2+} and His/Cys in aqueous solution and living cells

Lin Xu, Yufang Xu, Weiping Zhu, Buling Zeng, Chunmei Yang, Bin Wu and Xuhong Qian*

A versatile trifunctional chemosensor **RP** can selectively and sensitively recognize Cu^{2+} and Zn^{2+} in different solutions, respectively. Based on the zinc-containing $[\text{RP}@\text{Zn}^{2+}]$ complex, it shows highly selective recognition to His/Cys.

8288

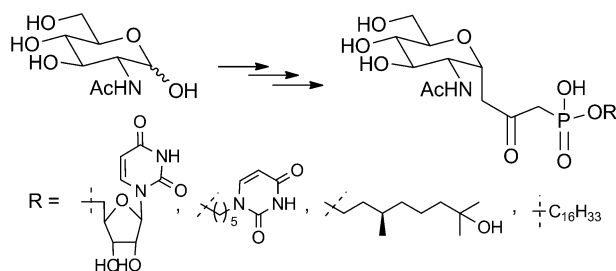


Synthesis and biological properties of conjugates between fluoroquinolones and a $\text{N}3'$ -functionalized pyochelin

Sabrina Noël, Véronique Gasser, Bénédicte Pesset, Françoise Hoegy, Didier Rognan, Isabelle J. Schalk and Gaëtan L. A. Mislin*

The binding and iron uptake properties of a $\text{N}3'$ -functionalized pyochelin are reported. This analog of a natural siderophore was used as a vector in the synthesis of unprecedented pyochelin–fluoroquinolone conjugates.

8301

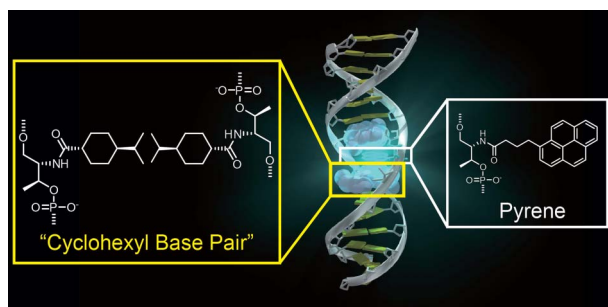


Synthesis and biological evaluation of potential new inhibitors of the bacterial transferase MraY with a β -ketophosphonate structure

Nicolas Auberger, Rok Frlan, Bayan Al-Dabbagh, Ahmed Bouhss, Muriel Crouvoisier, Christine Gravier-Pelletier* and Yves Le Merrer

Analogues of glycosyl nucleotides including a β -ketophosphonate structure, as pyrophosphate bioisostere, were synthesized and evaluated as MraY inhibitors.

8313



Cyclohexyl "base pairs" stabilize duplexes and intensify pyrene fluorescence by shielding it from natural base pairs

Hiromu Kashida,* Koji Sekiguchi, Naofumi Higashiyama, Tomohiro Kato and Hiroyuki Asanuma*

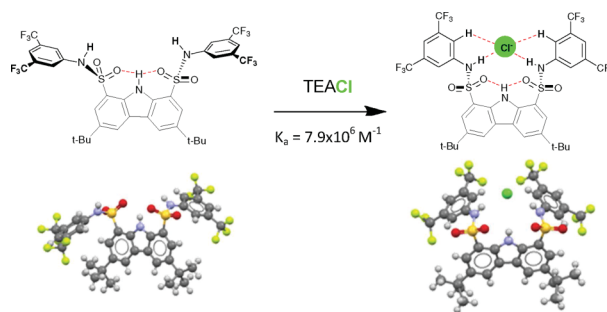
The stability and structure of novel artificial base pairs containing cyclohexyl rings were studied. These "base pairs", facilitated by hydrophobic interactions, showed even higher stability than natural A–T pairs. Their potential to prepare highly fluorescent labeling agents was demonstrated.

8321

Sulfonamide carbazole receptors for anion recognition

Ángel L. Fuentes de Arriba, María G. Turiel, Luis Simón, Francisca Sanz, Juan F. Boyero, Francisco M. Muñiz, Joaquín R. Morán and Victoria Alcázar*

The readily available sulfonamide carbazole receptors have been shown to have a higher affinity for halides over oxoanions. X-ray analysis of the complexes and modelling studies have been used to study this unusual preference.

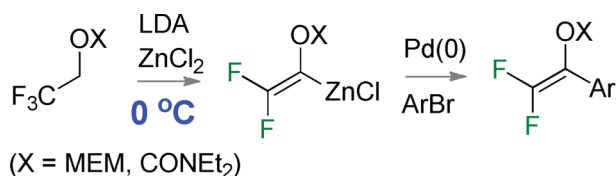


8328

One-pot near-ambient temperature syntheses of aryl(difluoroenol) derivatives from trifluoroethanol

Sara H. Kyne, Jonathan M. Percy,* Robert D. C. Pullin, Joanna M. Redmond and Peter G. Wilson

Difluoroalkenylzinc reagents prepared from trifluoroethanol derivatives at ice bath temperatures underwent convenient one-pot Negishi coupling with aryl halides.

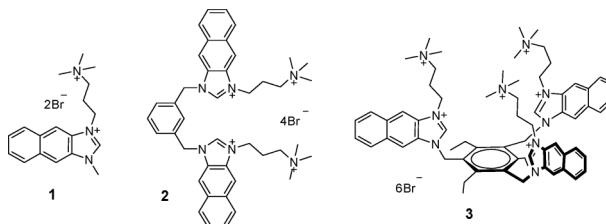


8340

Bis- and tris-naphthoimidazolium derivatives for the fluorescent recognition of ATP and GTP in 100% aqueous solution

Zhaochao Xu, Na Ri Song, Jong Hun Moon, Jin Yong Lee* and Juyoung Yoon*

In this paper, three naphthoimidazolium derivatives were synthesized and studied for the recognition of nucleotides.

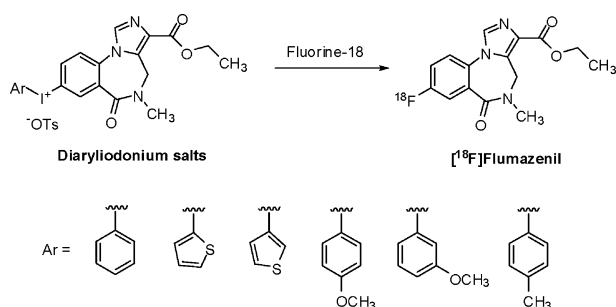


8346

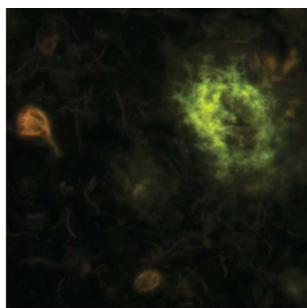
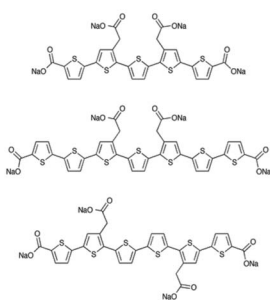
Facile aromatic radiofluorination of [¹⁸F]flumazenil from diaryliodonium salts with evaluation of their stability and selectivity

Byung Seok Moon, Hee Seup Kil, Jun Hyung Park, Ji Sun Kim, Jimin Park, Dae Yoon Chi, Byung Chul Lee* and Sang Eun Kim

Various conditions were explored using different diaryliodonium tosylate precursors with fluorine-18 for the efficient preparation of [¹⁸F]flumazenil.



8356

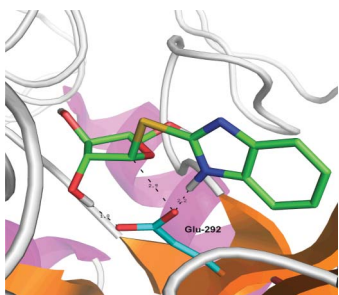


Synthesis of a library of oligothiophenes and their utilization as fluorescent ligands for spectral assignment of protein aggregates

Therèse Klingstedt, Andreas Åslund, Rozalyn A. Simon, Leif B. G. Johansson, Jeffrey J. Mason, Sofie Nyström, Per Hammarström and K. Peter R. Nilsson*

Anionic oligothiophenes comprising 5 to 7 thiophene units and carboxyl groups extending the conjugated backbone can be utilized as amyloid ligands for spectral assignment of pathological hallmarks observed in Alzheimer's disease.

8371

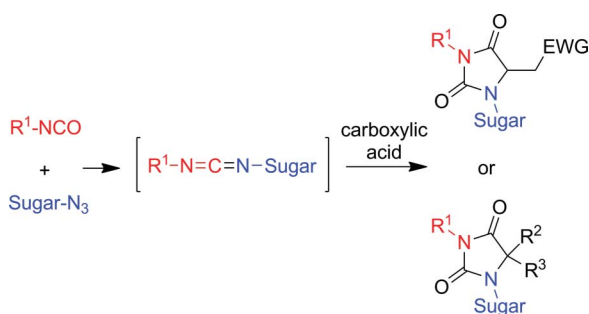


Exploring the synthetic potency of the first furanothiogliocoglycase through original remote activation

Mélanie Almendros, Dantcho Danalev, Marc François-Heude, Pascal Loyer, Laurent Legentil, Caroline Nugier-Chauvin, Richard Daniellou* and Vincent Ferrières*

Remote activation led to the enzymatic synthesis of various *S*-furanosides.

8379

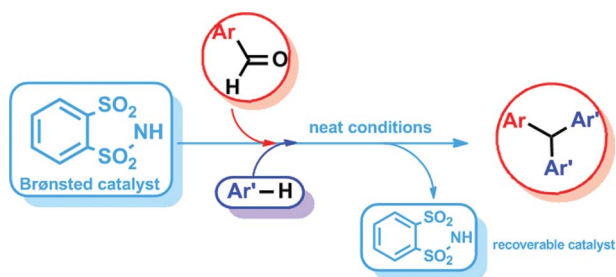


Three-component, one-pot sequential synthesis of glyco-hydantoin conjugates

Maria Cristina Bellucci, Alessandra Ghilardi and Alessandro Volonterio*

The development of a straightforward one-pot, three-component sequential process for linking sugar to hydantoins in very mild conditions and high yields is described.

8393



A new practical synthesis of triaryl and trisindolylmethanes under solvent-free reaction conditions

Margherita Barbero,* Silvano Cadamuro, Stefano Dughera, Claudio Magistris and Paolo Venturello

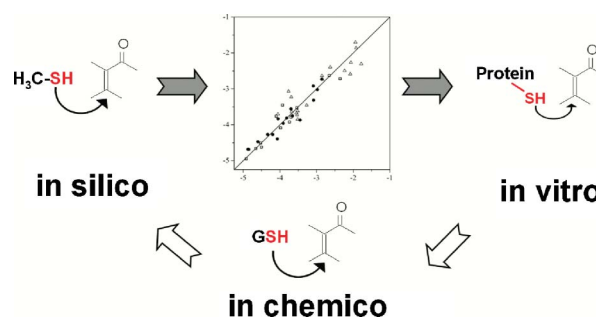
A new practical synthesis of triaryl and trisindolylmethanes is described under both homogeneous and heterogeneous catalysis using neat conditions.

8400

Predicting Michael-acceptor reactivity and toxicity through quantum chemical transition-state calculations

Denis Mulliner, Dominik Wondrousch and Gerrit Schüürmann*

Calculated transition-state energies of the reaction of Michael acceptors with methane thiol as model nucleophile inform about their aquatic toxicity.

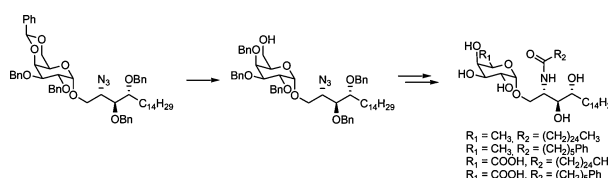


8413

Divergent synthetic approach to 6''-modified α -GalCer analogues

Nora Pauwels, Sandrine Aspeslagh, Gerd Vanhoenacker, Koen Sandra, Esther D. Yu, Dirk M. Zajonc, Dirk Elewaut, Bruno Linclau and Serge Van Calenbergh*

We present a practical synthetic route for the synthesis of galacturonic acid and D-fucosyl modified KRN7000, featuring the regioselective opening of a 4'',6''-O-benzylidene ring as the key step.

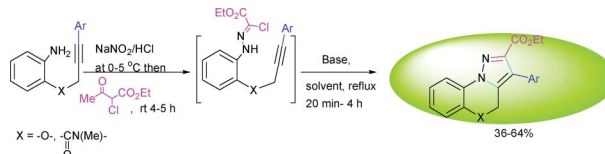


8422

An efficient strategy for the general synthesis of 3-aryl substituted pyrazolo[5,1-c][1,4]benzoxazines and pyrazolo[1,5-a][1,4]benzodiazepin-6(4H)-ones

Kaushik Brahma, Anup Kumar Sasmal and Chinmay Chowdhury*

A facile method has been developed for the general synthesis of 3-aryl substituted pyrazolo[5,1-c][1,4]benzoxazines and pyrazolo[1,5-a][1,4]benzodiazepin-6(4H)-ones.

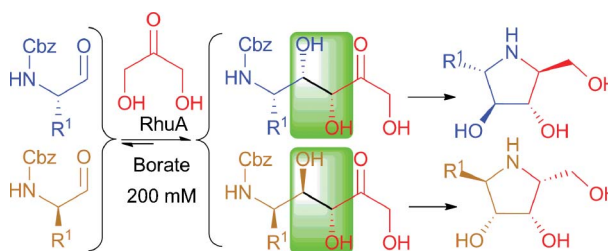


8430

Highly efficient aldol additions of DHA and DHAP to N-Cbz-amino aldehydes catalyzed by L-rhamnulose-1-phosphate and L-fuculose-1-phosphate aldolases in aqueous borate buffer

Xavier Garrabou, Jordi Calveras, Jesús Joglar, Teodor Parella, Jordi Bujons and Pere Clapés*

A two step strategy, combining an enzymatic aldol addition and reductive amination, for an efficient and expedient preparation of iminocyclitols.



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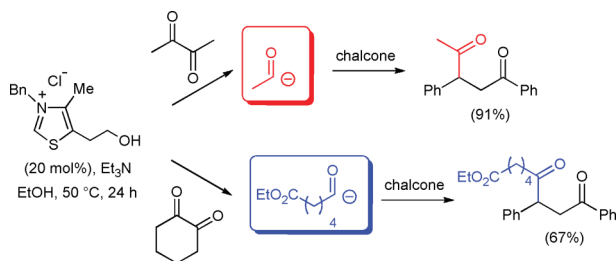
Closing date for nominations is
15 January 2012

8437

Thiazolium-catalyzed intermolecular Stetter reaction of linear and cyclic alkyl α -diketones

Olga Bortolini, Giancarlo Fantin, Marco Fogagnolo, Pier Paolo Giovannini, Alessandro Massi* and Salvatore Pacifico

Alkyl 1,2-diketones serve as a novel source of acyl anions for intermolecular Stetter reactions.

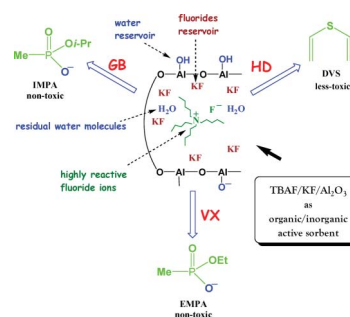


8445

The reactivity of quaternary ammonium- versus potassium-fluorides supported on metal oxides: paving the way to an instantaneous detoxification of chemical warfare agents

Yossi Zafrani,* Lea Yehezkel, Michael Goldvaser, Daniele Marciano, Daniel Waysbort, Eytan Gershonov and Ishay Columbus*

A new organic/inorganic solid support TBAF/KF/Al₂O₃ is a powerful active sorbent for the detoxification of the chemical warfare agents VX, GB and HD, due to a synergistic effect of the two types of fluorides.

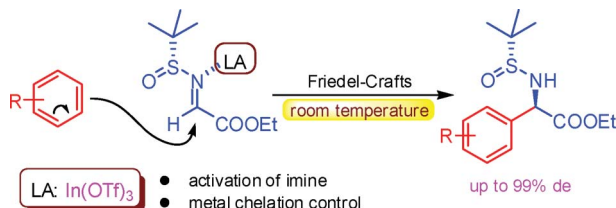


8452

Highly diastereoselective Friedel–Crafts reaction of arenes with *N*-*tert*-butanesulfinylimino ester towards the efficient synthesis of α -arylglycines

Yi Li, Du-Ming Ji and Ming-Hua Xu*

Lewis acid-catalyzed highly diastereoselective asymmetric Friedel–Crafts alkylation of arenes with a chiral *N*-*tert*-butanesulfinylimino ester is described. The reaction allows exceptionally mild, efficient and ready access to various enantiomerically enriched (up to 99% de) α -arylglycines.

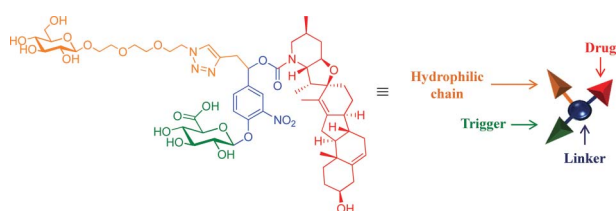


8459

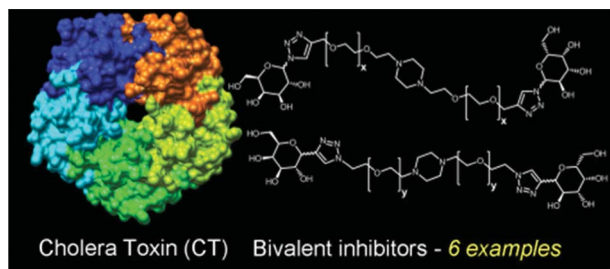
A new cycloamine glucuronide prodrug with improved kinetics of drug release

Brigitte Renoux, Thibaut Legigan, Souheyla Bensalma, Corinne Chadéneau, Jean-Marc Muller and Sébastien Papot*

A hydrophilic glucuronide prodrug of cycloamine designed to target selectively the Hedgehog signalling pathway of cancer cells was studied.



8465

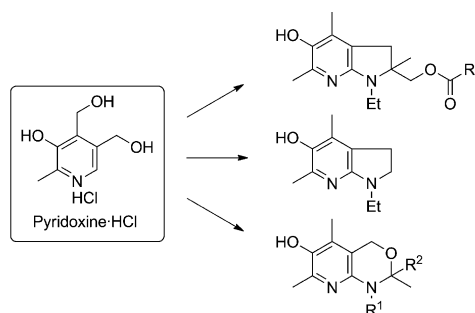


Synthesis of 1,2,3-triazole linked galactopyranosides and evaluation of cholera toxin inhibition

David J. Leaver, Raymond M. Dawson, Jonathan M. White, Anastasios Polyzos* and Andrew B. Hughes*

Synthesis of bivalent anomericly pure α - and β -configured, C- and N-linked galactopyranosyl triazolyl inhibitors of cholera toxin (CT) is described.

8475

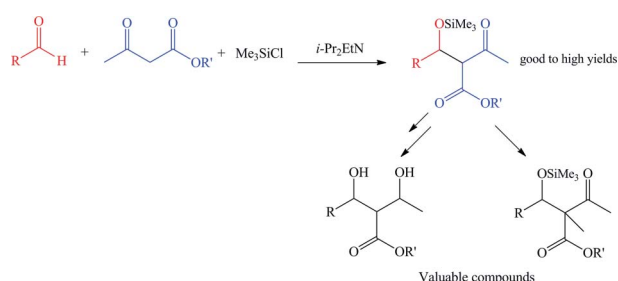


Pyridoxine-derived bicyclic aminopyridinol antioxidants: synthesis and their antioxidant activities

Tae-gyu Nam, Jin-Mo Ku, Christopher L. Rector, Hoyoung Choi, Ned A. Porter* and Byeong-Seon Jeong*

A few facile synthetic pathways for bicyclic aminopyridinol antioxidants are presented. Attachment of a long alkyl chain to the bicyclic pyridinol scaffold was established using ester linkage. Non-substituted pyrrolopyridinols and 1,3-oxazine-fused pyridinols were also synthesized as novel antioxidant scaffolds. Antioxidant activities were measured by a radical clock method.

8483

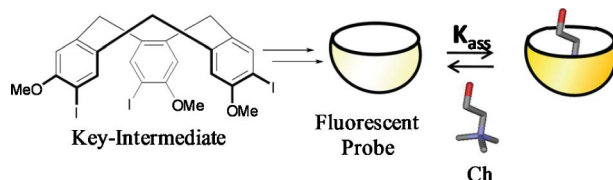


Multi-component, regio-selective aldol addition of β -ketoesters to aldehydes: scope and applications

Vijaykumar More, Antonia Di Mola, Gianluca Croce, Consiglia Tedesco, Carmen Petronzi, Antonella Peduto, Paolo De Caprariis, Rosanna Filosa and Antonio Massa*

Simple and effective multi-component aldol reactions of β -ketoesters to a series of aldehydes is described.

8489



C₃-triiodocyclotrimeratrylene as a key intermediate to fluorescent probes: application to selective choline recognition

Lisa Peyrard, Sabine Chierici, Sandra Pinet, Pinar Batat, Gediminas Jonusauskas, Noël Pinaud, Pierre Meyrand and Isabelle Gosse*

The potential of iodinate cyclotrimeratrylene is illustrated through the synthesis of a fluorescent probe binding choline in pseudo-physiological conditions.

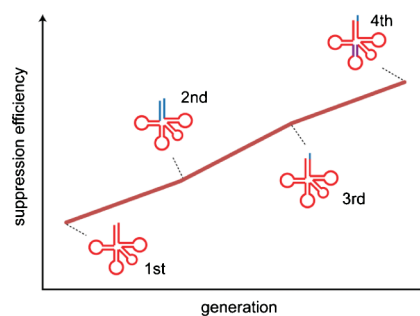
PAPERS

8495

Improvement of *in vitro*-transcribed amber suppressor tRNAs toward higher suppression efficiency in wheat germ extract

Atsushi Ogawa,* Yasunori Doi and Nobuto Matsushita

In vitro-transcribed, unmodified, and non-aminoacylated amber suppressor tRNAs were optimized toward higher suppression efficiency in wheat germ extract.



ADDITIONS AND CORRECTIONS

8504

Additions and corrections published in 2011.

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