

Organic & Biomolecular Chemistry

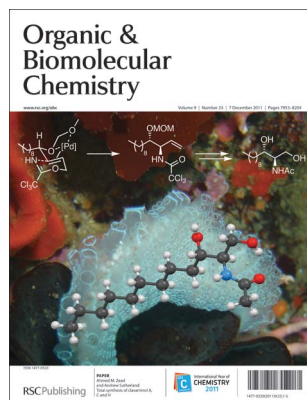
An international journal of synthetic, physical and biomolecular organic chemistry

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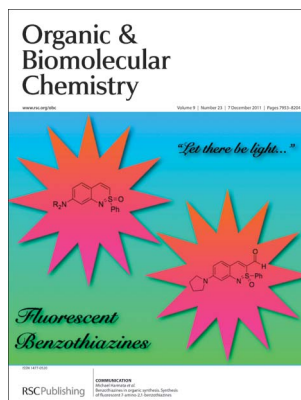
ISSN 1477-0520 CODEN OBCRAK 9(23) 7953–8204 (2011)



Cover

Ahmed M. Zead and Andrew Sutherland, pp. 8030–8037. Total synthesis of clavaminol A, C and H, using an ether directed palladium(II)-catalysed Overman rearrangement. Background photograph reproduced with permission from Peter Southwood, Southern Underwater Research Group (SURG).

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Inside cover

See Michael Harmata *et al.*, pp. 7979–7982.

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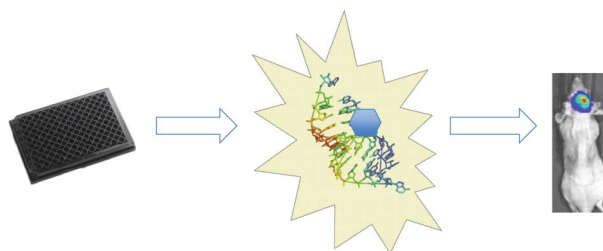
PERSPECTIVE

7969

Development and utilization of non-coding RNA–small molecule interactions

Wesleigh E. Georgianna and Douglas D. Young*

Small molecule interactions with non-coding RNAs represent a novel target for therapeutic intervention, requiring the development of robust screens to better understand these interactions and develop new drugs.



COMMUNICATIONS

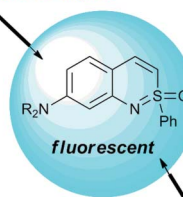
7979

Benzothiazines in organic synthesis. Synthesis of fluorescent 7-amino-2,1-benzothiazines

Nattawut Yongpruksa, Siddharth Pandey, Gary A. Baker and Michael Harmata*

Fluorescent 7-amino-2,1-benzothiazines were prepared in high yields using the palladium-catalyzed reaction of 4-amino-2-chlorobenzaldehydes with a sulfoximine or the reaction of 7-fluoro-2,1-benzothiazines with amines.

N-Arylation of sulfoximine with 2-chloro-4-aminobenzaldehyde



SnAr between amine and 7-Fluorobenzothiazine

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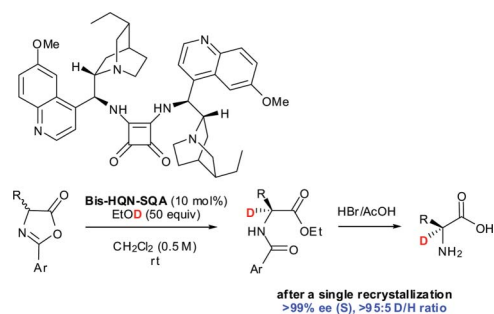
COMMUNICATIONS

7983

Enantioselective synthesis of α -deuterium labelled chiral α -amino acids *via* dynamic kinetic resolution of racemic azlactones

Joong-Suk Oh, Kyung Il Kim and Choong Eui Song*

Catalytic dynamic kinetic resolution of racemic azlactones with EtOD using squaramide-based dimeric cinchona alkaloid organocatalysts is shown to be an effective strategy for the preparation of enantiomerically pure α -deuterated α -amino acids.

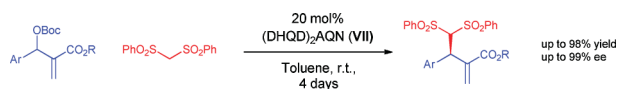


7986

Enantioselective organocatalytic asymmetric allylic alkylation. Bis(phenylsulfonyl)methane addition to MBH carbonates

Xavier Companyó, Guillem Valero, Victor Ceban, Teresa Calvet, Mercé Font-Bardía, Albert Moyano* and Ramon Rios*

The organocatalytic allylic substitution of MBH carbonates with bis(phenylsulfonyl)methane was simply catalyzed by (DHQD)₂AQN affording the final compounds in good yields and enantioselectivities.

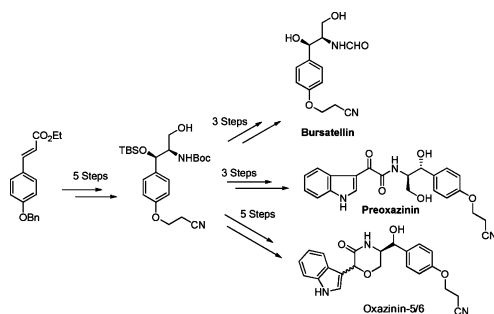


7990

Asymmetric first total syntheses and assignment of absolute configuration of oxazinin-5, oxazinin-6 and preoxazinin-7

Dattatraya H. Dethe,* Alok Ranjan and Vijendra H. Pardeshi

Asymmetric first total syntheses of the toxins oxazinin-5, oxazinin-6 and preoxazinin-7 have been achieved from a common intermediate, derived from a regiocontrolled Sharpless asymmetric aminohydroxylation and oxa-Michael reaction, which in addition to confirming their structures also established their absolute configuration. An expeditious synthesis of metabolite bursatellin was also completed.

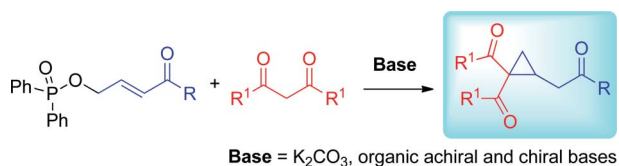


7993

Base promoted synthesis of activated cyclopropanes bearing homologated carbonyl groups *via* tandem Michael addition–intramolecular enolate trapping

Alessio Russo and Alessandra Lattanzi*

Novel cyclopropanes bearing homologated carbonyl groups were obtained by exploiting a base-promoted MIRC reaction featuring intramolecular enolate trapping of 1,3-dicarbonyl compounds with γ -hydroxyenone derived diphenyl phosphinates.



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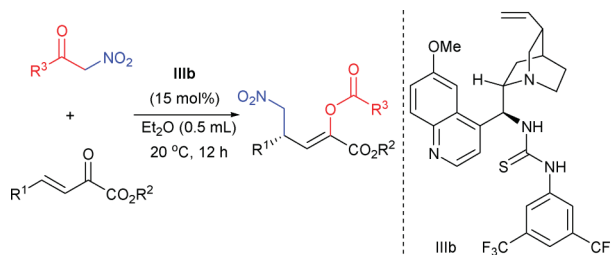
COMMUNICATIONS

7997

Organocatalytic asymmetric Michael-type reaction between β,γ -unsaturated α -keto ester and α -nitro ketone

Pengfei Li, Sau Hing Chan, Albert S. C. Chan* and Fuk Yee Kwong*

Michael-type reaction between β,γ -unsaturated α -keto ester and α -nitro ketone resulted in 47–94% yield and 68–96% ee, which offered efficient access to chiral 5-nitro-pent-2-enoates, a precursor to chiral α -ketolactam.

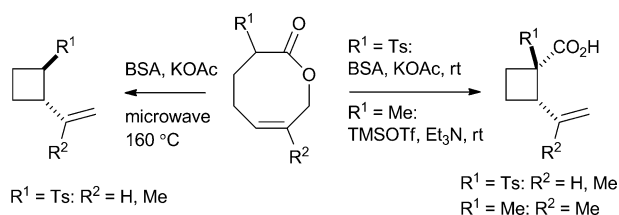


8000

Transannular Claisen rearrangement reactions for the synthesis of vinylcyclobutanes: formal synthesis of (\pm)-grandisol

Donald Craig,* Kiyohiko Funai, Sophie J. Gore, Albert Kang and Alexander V. W. Mayweg

Unsaturated eight-membered lactones undergo decarboxylative and non-decarboxylative transannular Ireland–Claisen rearrangement reactions, to give substituted vinylcyclobutanes. A formal synthesis of (\pm)-grandisol is described.

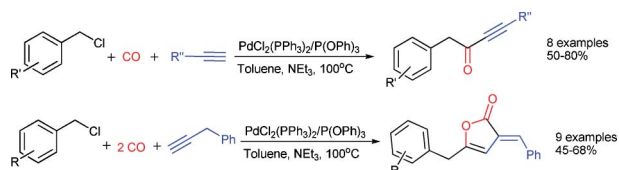


8003

Palladium-catalyzed carbonylative coupling of benzyl chlorides with terminal alkynes to give 1,4-diaryl-3-butyne-2-ones and related furanones

Xiao-Feng Wu, Helfried Neumann and Matthias Beller*

Palladium-catalyzed carbonylative coupling of benzyl chlorides with terminal alkynes to give the corresponding alkyneones and furanones has been developed. 8 different alkyneones and 9 different furanones were prepared in moderate yields.



PAPERS

8006

Tuning the activity of glutathione peroxidase mimics through intramolecular Se \cdots N,O interactions: A DFT study incorporating solvent-assisted proton exchange (SAPE)

Craig A. Bayse* and Andrea Pavlou

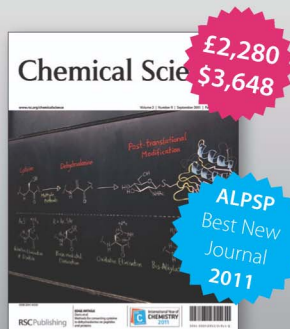
DFT-SAPE studies using explicit water molecules to facilitate proton exchange in gas-phase models show that the GPx-like activity of ortho-substituted aryl selenols depends on the ease of displacement of the Se \cdots N,O donor–acceptor interaction.



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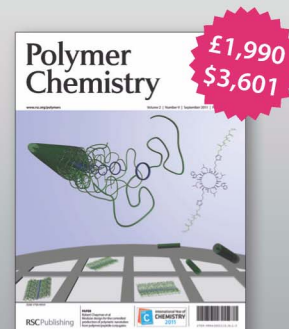
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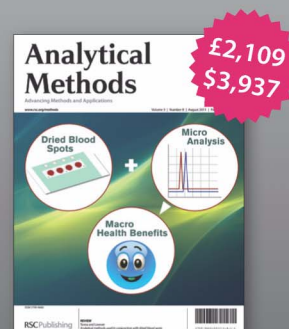
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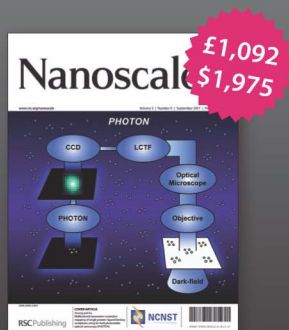
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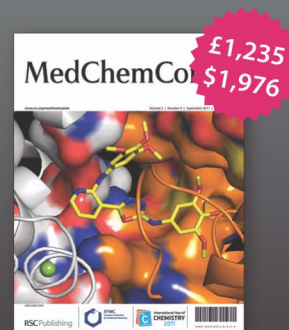
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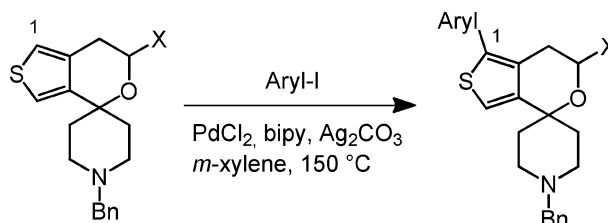
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8016

Exploitation of an additional hydrophobic pocket of σ_1 receptors: Late-stage diverse modifications of spirocyclic thiophenes by C–H bond functionalization

Christina Meyer, Benedikt Neue, Dirk Schepmann, Shuichi Yanagisawa, Junichiro Yamaguchi, Ernst-Ulrich Würthwein, Kenichiro Itami* and Bernhard Wünsch*

Selective α -arylation of complex spirocyclic thiophenes with further functional groups allows the exploitation of a hydrophobic pocket of σ_1 receptors.

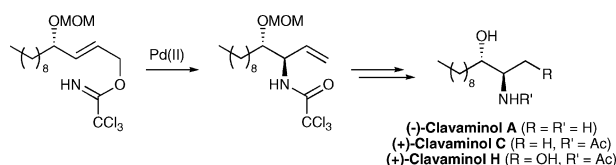


8030

Total synthesis of clavaminol A, C and H

Ahmed M. Zaed and Andrew Sutherland*

The first total synthesis of clavaminol A, C and H has been achieved using a palladium(II)-catalysed directed Overman rearrangement to create the key C–N bond and install the *erythro* configuration.

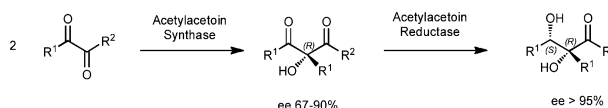


8038

Enzymatic diastereo- and enantioselective synthesis of α -alkyl- α,β -dihydroxyketones

Pier Paolo Giovannini,* Giancarlo Fantin, Alessandro Massi, Valentina Venturi and Paola Pedrini

Optically pure α -alkyl- α,β -dihydroxyketones are synthesized from α -diketones through an enzymatic two-step procedure.

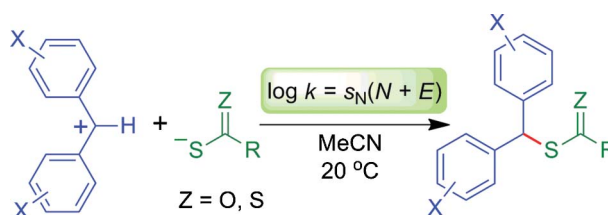


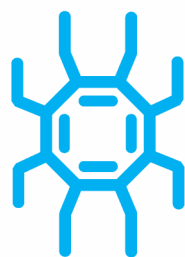
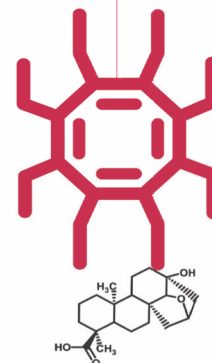
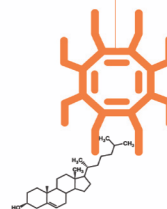
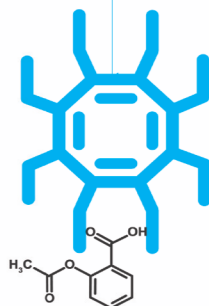
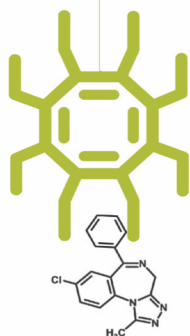
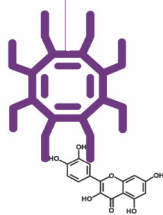
8046

Characterization of the nucleophilic reactivities of thiocarboxylate, dithiocarbonate and dithiocarbamate anions

Xin-Hua Duan, Biplab Maji and Herbert Mayr*

The kinetics of the reactions of thiocarboxylate and thiocarbonate ions with laser-flash photolytically generated benzhydrylium ions were determined to derive reactivity parameters N (and s_N) for these types of nucleophiles.





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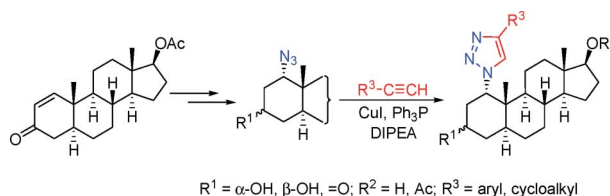
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8051

Efficient approach to novel 1 α -triazolyl-5 α -androstane derivatives as potent antiproliferative agents

Zalán Kádár, Ádám Baji, István Zupkó, Tibor Bartók, János Wölfling and Éva Frank*

Novel 1 α -azides underwent 1,3-dipolar cycloaddition with terminal alkynes in the presence of Cu(I)-catalyst to afford *exo*-triazolyl derivatives in good to excellent yields.

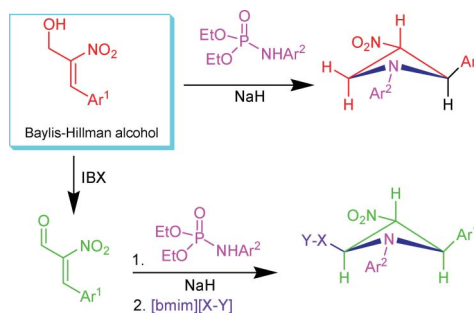


8058

Strategic applications of Baylis–Hillman adducts to general syntheses of 3-nitroazetidines

Ankita Rai and Lal Dhar S. Yadav*

A highly diastereoselective annulation of Baylis–Hillman alcohols and their aldehydes with *N*-aryl/tosylphosphoramidates affords 3-nitroazetidines under mild conditions.

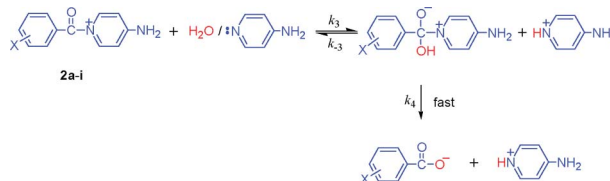


8062

Hydrolysis of 1-(*X*-substituted-benzoyl)-4-aminopyridinium ions: effect of substituent *X* on reactivity and reaction mechanism

Ik-Hwan Um,* Eun-Hee Kim and Ji-Sun Kang

Hydrolysis of **2a–i** proceeds through a stepwise mechanism with formation of an intermediate being rate limiting.

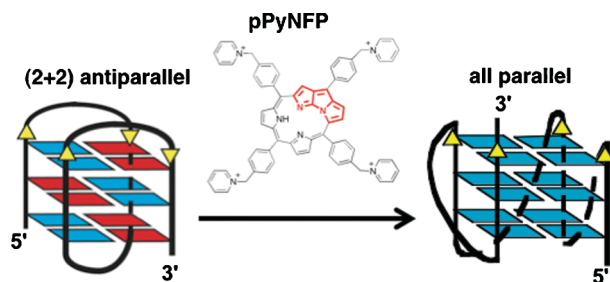


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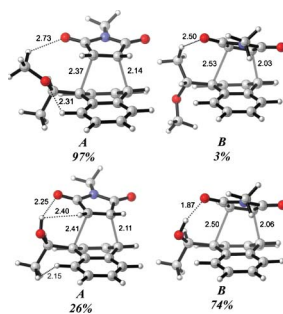
N-fused porphyrin with pyridinium side-arms: a new class of aromatic ligand with DNA-binding ability

Yoshiya Ikawa, Satoshi Touden and Hiroyuki Furuta*

A water-soluble derivative of *N*-fused porphyrin (NFP) modulates G-quadruplex DNA structure.



8079

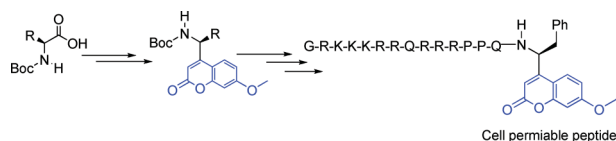


Origins of the diastereoselectivity in hydrogen bonding directed Diels–Alder reactions of chiral dienes with achiral dienophiles: a computational study

Sesil Agopcan, Nihan Çelebi-Ölçüm, Melek Nihan Üçışık, Amitav Sanyal and Viktorya Aviyente*

The origins of diastereoselectivity in the H-bonding assisted Diels–Alder reactions of chiral dienes with achiral dienophiles was investigated with the distortion/interaction model.

8089

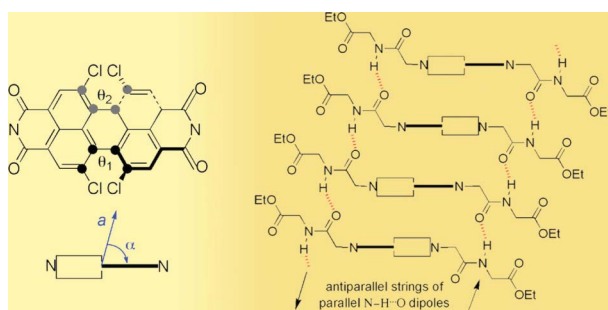


A facile transformation of amino acids to functionalized coumarins

Anupam Bandyopadhyay and Hosahudya N. Gopi*

A facile, efficient and racemization-free transformation of amino acids to functionalized coumarins, and their incorporation into a cell permeable peptide, are described.

8096

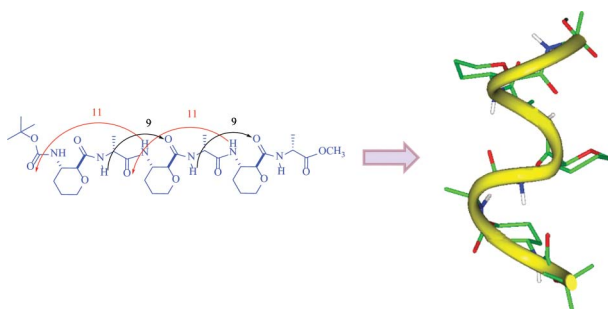


Amino acid derivatives of perylenediimide and their N–H...O peptide bond dipoles-templated solid state assembly into stacks

Cyprien Lemouchi, Sergey Simonov, Leokadiya Zorina, Christelle Gautier, Pierrick Hudhomme and Patrick Batail*

The constraint inherent to large collections of N–H...O hydrogen bond parallel electric dipoles running alongside stacks reduces the dihedral angles around the bay regions by as much as 11% down to 32° in a series of crystalline peptide-appended perylenediimides.

8102



Design and synthesis of *trans*-3-aminopyran-2-carboxylic acid (APyC) and α/β -peptides with 9/11-helix

Gangavaram V. M. Sharma,* Kodeti Srinivas Reddy, Shaik Jeelani Basha, Kondreddi Ravinder Reddy and Akella V. S. Sarma*

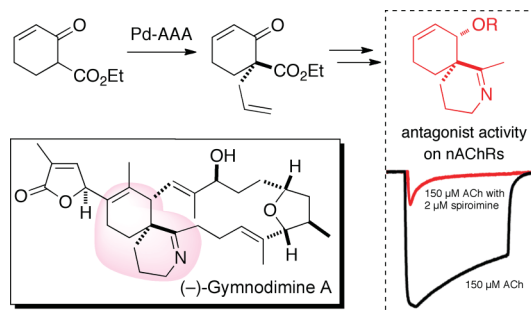
The participation of pyran oxygen of *trans*-3-aminopyran-2-carboxylic acid (APyC) in α/β -peptides resulting in 9/11-helix is envisaged to provide scope for new designs.

8112

6,6-Spiroimine analogs of (–)-gymnodimine A: synthesis and biological evaluation on nicotinic acetylcholine receptors

Leslie Duroure, Thierry Jousseume, Rómulo Aráoz, Elvina Barré, Pascal Retailleau, Laurent Chabaud,* Jordi Molgó* and Catherine Guillou*

Synthetic spiroimine analogs of (–)-gymnodimine A inhibit nicotinic acetylcholine receptors, but are less potent than the natural toxin.

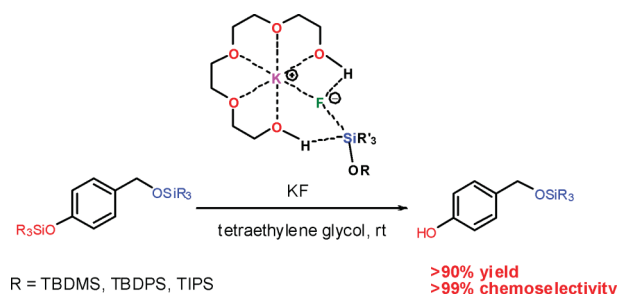


8119

A mild and efficient method for the selective deprotection of silyl ethers using KF in the presence of tetraethylene glycol

Hailong Yan, Joong-Suk Oh and Choong Eui Song*

A mild and efficient protocol for the selective deprotection of silyl ethers using KF in an oligoethene glycol (*e.g.*, tetraethylene glycol) is reported.

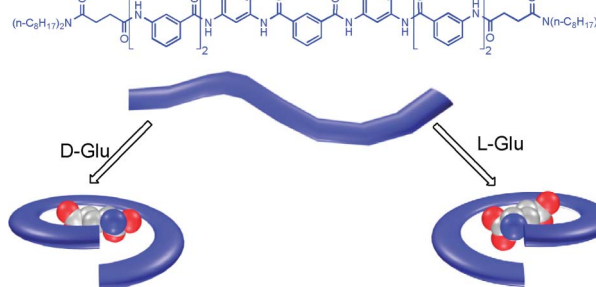


8122

meta-Substituted benzamide oligomers that complex mono-, di- and tricarboxylates: folding-induced selectivity and chirality

Zhu-Ming Shi, Shi-Gui Chen, Xin Zhao,* Xi-Kui Jiang and Zhan-Ting Li*

Benzamide-derived oligomers fold into compact conformations to complex aromatic and aliphatic carboxylate anions through multiple intermolecular N–H \cdots O and C–H \cdots O hydrogen bonds, and display helical chirality upon binding chiral glutamic acid dianion.

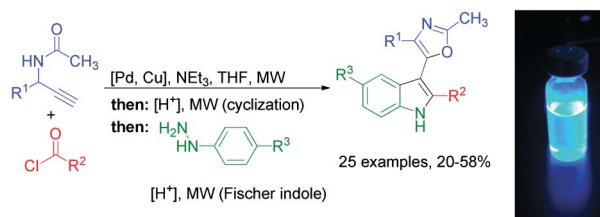


8130

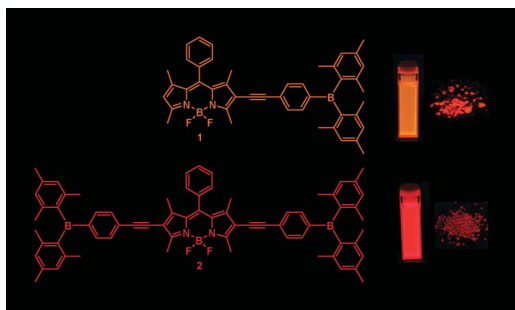
Blue-luminescent 5-(3-indolyl)oxazoles via microwave-assisted three-component coupling–cycloisomerization–Fischer indole synthesis

Oliver Grotkopp, Atia Ahmad, Walter Frank and Thomas J. J. Müller*

Blue luminescent aryl substituted 5-(3-indolyl)oxazoles are readily synthesized in a novel one-pot three-component microwave assisted synthesis consisting of a sequence of Sonogashira coupling, an acid-catalyzed cycloisomerization, and a concluding Fischer indole synthesis.



8141

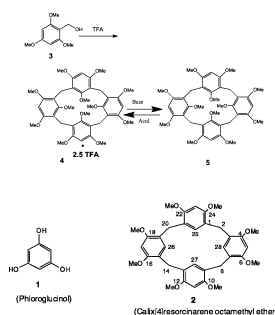


Solid-state emissive triarylborane-based BODIPY dyes: Photophysical properties and fluorescent sensing for fluoride and cyanide ions

Guang-Liang Fu, Hong Pan, Yi-Hong Zhao and Cui-Hua Zhao*

The solid-state emissive BODIPY dyes were achieved by introduction of the bulky substituent, [(4-dimesitylboryl)phenyl]ethynyl at the 2- and 2,6-positions.

8147

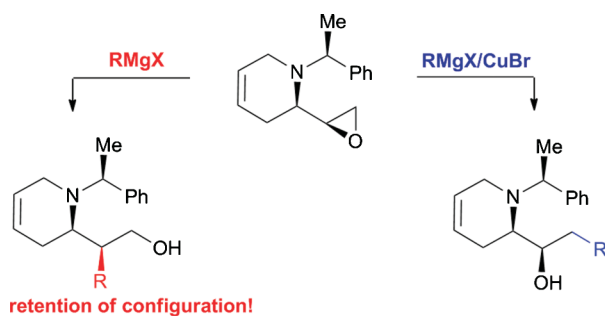


Synthesis, structure and spectroscopic properties of calix[4]phloroglucinarene dodecamethyl ether and its trifluoroacetic acid complex

Olusegun M. Falana,* Philip M. Keehn* and Robert Stevenson

A novel family of metacyclophanes, monikered phloroglucinarene, in free and acid-complex states is presented. The stereochemistry and electronic properties are reported permitting the first understanding of the influence of intercavity moieties on conformation in the liquid phase.

8155

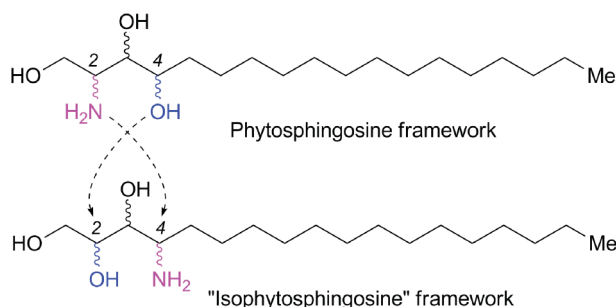


Switch in regioselectivity of epoxide ring-opening by changing the organometallic reagent

José A. Gálvez,* María D. Díaz de Villegas,* Ramón Badorrey and Pilar López-Ram-de-Viú

An efficient entry to piperidines with a hydroxyalkyl substituent at C2 by epoxide ring opening of a 2-(2'-oxiranyl)piperidine is reported. Regioselectivity in the nucleophilic attack is controlled by the organometallic reagent.

8163



Asymmetric synthesis and cytotoxic activity of isomeric phytosphingosine derivatives

Arnaud Rives, Cécile Baudoin-Dehoux, Nathalie Saffon, Nathalie Andrieu-Abadie and Yves Génisson*

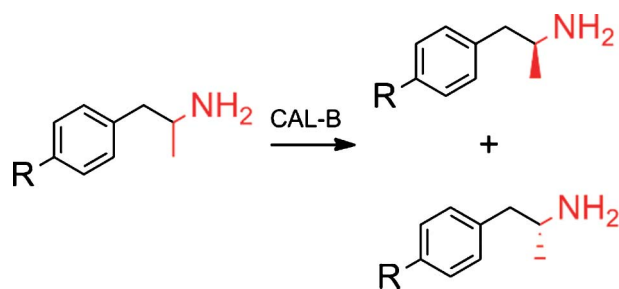
Phytosphingosine analogues embedding a substitution pattern resulting from a permutation of the C-2 and C-4 substituents along the sphingoid base skeleton were conceived, synthesised and tested regarding their cytotoxicity.

8171

Enzymatic enantiomeric resolution of phenylethylamines structurally related to amphetamine

Lourdes Muñoz, Anna M. Rodriguez, Gloria Rosell, M. Pilar Bosch and Angel Guerrero*

Both enantiomers of several phenylethylamines, structurally related to amphetamine, have been prepared in good yields and excellent enantiomeric purity by enzymatic kinetic resolution.

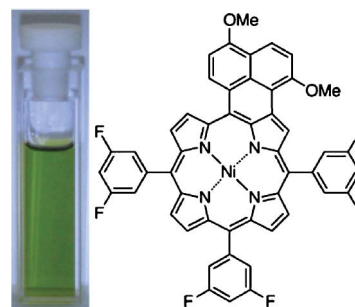


8178

Naphthalene-fused metallo-porphyrins—synthesis and spectroscopy

Jan P. Lewtak, Dorota Gryko, Duoduo Bao, Ernest Sebai, Olena Vakuliuk, Mateusz Ścigaj and Daniel T. Gryko*

Copper and nickel porphyrin-complexes were successfully transformed using $\text{Fe}(\text{ClO}_4)_3 \cdot 2\text{H}_2\text{O}$ into π -expanded porphyrins in 40–83% yield.

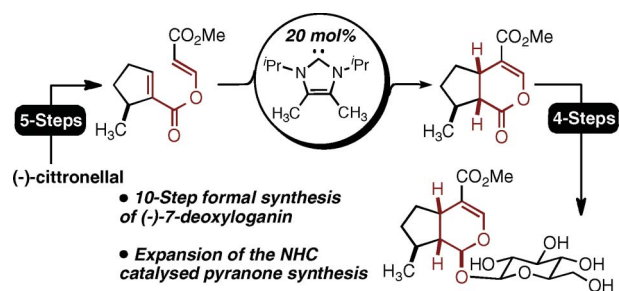


8182

Concise formal synthesis of (–)-7-deoxyloganin via *N*-heterocyclic carbene catalysed rearrangement of α,β -unsaturated enol esters

Lisa Candish and David W. Lupton*

NHC catalysed rearrangement of α,β -unsaturated enol esters provides the cyclopentapyranone core of (–)-7-deoxyloganin (1), with diastereo- and chemoselectivity in 5-steps starting from (–)-citronellal, and enables completion of a formal total synthesis in 10-steps.

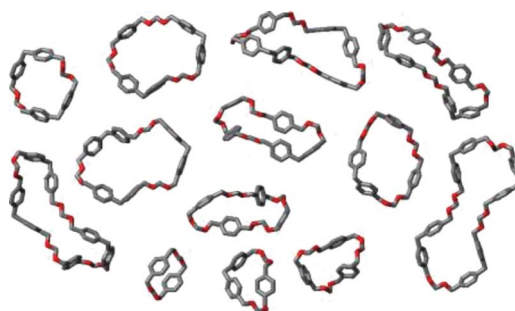


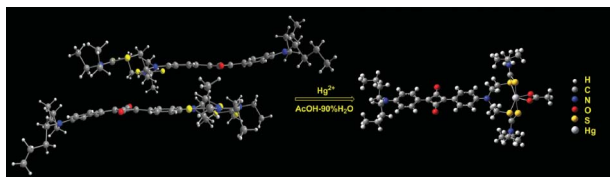
8190

A well-behaved dynamic library of cyclophane formaldehyde acetals incorporating diphenylmethane units

Josè Augusto Berrocal, Roberta Cacciapaglia and Stefano Di Stefano*

The investigation of a well-behaved DL based on acetal cyclophanes incorporating diphenylmethane units is reported.





Dual-mode unsymmetrical squaraine-based sensor for selective detection of Hg^{2+} in aqueous media

Cheng Chen, Haijun Dong, Yaqing Chen, Liangqia Guo, Zhenyu Wang, Jian-Jun Sun and Nanyan Fu*

A novel “turn-on” fluorescent and colorimetric chemosensor based on unsymmetrical squaraine dye (USQ-1) for the selective detection of Hg^{2+} in aqueous media is described, and a recognition mechanism *via* the Hg^{2+} -induced deaggregation of the dye molecule based on the binding mode is proposed.

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