

Organic & Biomolecular Chemistry

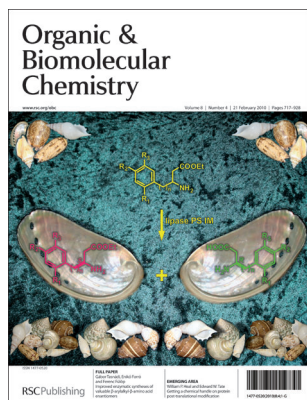
An international journal of synthetic, physical and biomolecular organic chemistry

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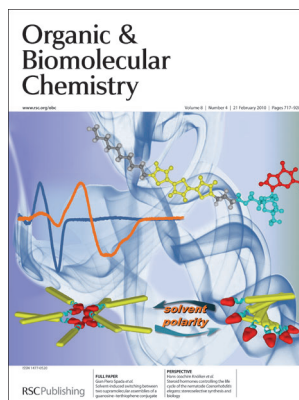
ISSN 1477-0520 CODEN OBCRAK 8(4) 717–928 (2010)



Cover

See Ferenc Fülöp *et al.*, pp. 793–799.
The enantioselective *Burkholderia cepacia*-catalysed hydrolyses of biologically relevant β -arylalkyl-substituted β -amino esters were performed with high enantiomeric excesses and in good yields.

Image reproduced by permission of Gábor Tasnádi, Enikő Forró and Ferenc Fülöp from *Org. Biomol. Chem.*, 2010, **8**, 793.



Inside cover

See Gian Piero Spada *et al.*, pp. 774–781.
Guanine or terthienyl: which one leads the self-assembly of the guanosine–terthiophene conjugate? Upon increasing solvent polarity, the supramolecular aggregate directed by guanine stacking is reversibly converted into a terthienyl stacked architecture.

Image reproduced by permission of Gian Piero Spada *et al.* from *Org. Biomol. Chem.*, 2010, **8**, 774.

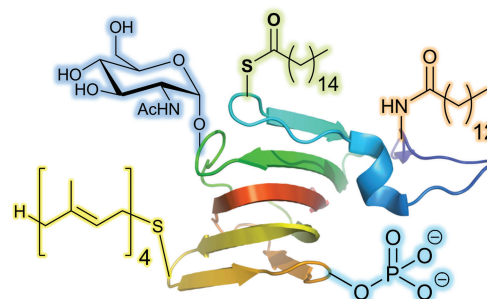
EMERGING AREA

731

Getting a chemical handle on protein post-translational modification

William P. Heal and Edward W. Tate*

Chemical proteomics is a powerful technology for the study of post- and co-translational modification of proteins. Here, we review techniques that combine protein-modifying enzymes with bioorthogonal chemoselective elaboration to enable new advances in our understanding of protein modification.



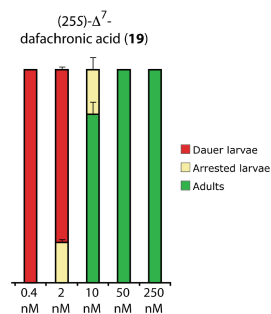
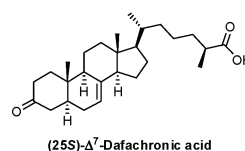
PERSPECTIVE

739

Steroid hormones controlling the life cycle of the nematode *Caenorhabditis elegans*: stereoselective synthesis and biology

René Martin, Eugeni V. Entchev, Teymuraz V. Kurzchalia and Hans-Joachim Knölker*

Cholesterol-derived hormones, the dafachronic acids, play a major role in controlling the life cycle and initiating dauer larva formation of the nematode *Caenorhabditis elegans*. This Perspective describes recent progress in the synthesis of these steroid hormones and their biological function.



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Organic & Biomolecular Chemistry (print: ISSN
1477-0520; electronic: ISSN 1477-0539) is published 24 times
a year by the Royal Society of Chemistry, Thomas Graham
House, Science Park, Milton Road, Cambridge, UK CB4 0WF.
All orders, with cheques made payable to the
Royal Society of Chemistry, should be sent to RSC
Distribution Services, c/o Portland Customer Services,
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2010 Annual (print+electronic) subscription price: £3105;
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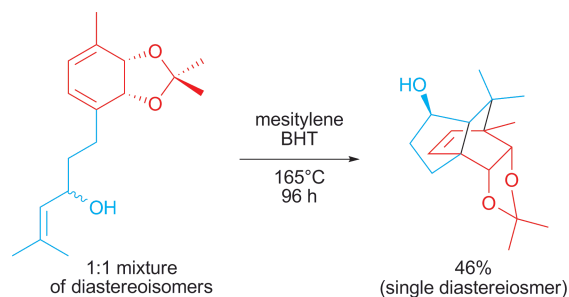
COMMUNICATIONS

751

Chemoenzymatic and enantiodivergent routes to 1,2-ring-fused bicyclo[2.2.2]octane and related tricyclic frameworks

Kerrie A. B. Austin, Jon D. Elsworth, Martin G. Banwell* and Anthony C. Willis

The chemoenzymatically derived triene undergoes a facially-selective IMDA reaction to give an adduct incorporating the carbocyclic framework of the sesquiterpene (–)-khusiol.

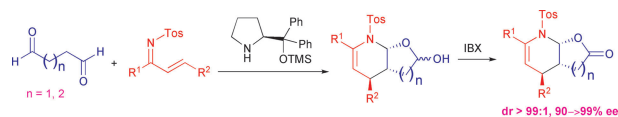


755

Enantioselective construction of lactone[2,3-*b*]piperidine skeletons *via* organocatalytic tandem reactions

Zhao-Quan He, Bo Han, Rui Li,* Li Wu and Ying-Chun Chen*

A highly enantioselective construction of δ - and γ -lactone[2,3-*b*]piperidine skeletons was accomplished by tandem aza-Diels–Alder reaction–hemiacetal formation–oxidation from *N*-Tos-1-aza-1,3-butadienes and aliphatic dialdehydes.

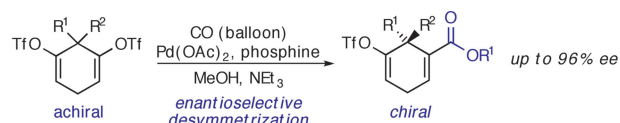


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Enantioselective desymmetrizing palladium catalyzed carbonylation reactions: the catalytic asymmetric synthesis of quaternary carbon center containing 1,3-dienes

Simon J. Byrne, Anthony J. Fletcher, Paul Hebeisen and Michael C. Willis*

A desymmetrization protocol has been used to develop a palladium catalyzed enantioselective carbonylation process. Achiral cyclic bis-alkenyltriflates are converted to their corresponding monoester derivatives with selectivities of up to 96% ee.

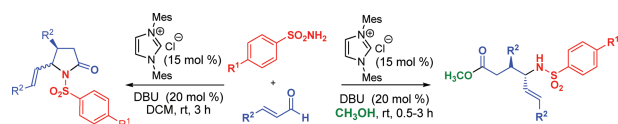


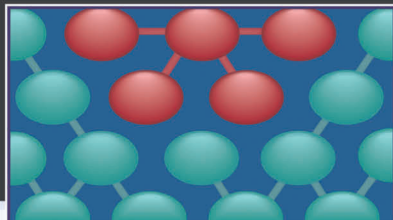
761

A novel pseudo four component reaction involving homoenolate for the synthesis of γ -aminobutyric acid (GABA) derivatives

Vijay Nair,* Vimal Varghese, Beneesh P. Babu, C. R. Sinu and Eringathodi Suresh

Homoenolate generated from α,β -unsaturated aldehydes by NHC catalysis underwent facile addition to conjugated sulfonimines and subsequent methanolysis to afford protected GABA derivatives stereoselectively and in high yields, thus constituting a novel pseudo four component reaction.





MedChem Europe

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Gerhard Mueller, Chief Scientific Officer, Proteros Fragments

Johannes Kirchmair, Assistant Professor, University of Innsbruck

Artem Cherkasov, Assistant Professor, University of British Columbia

Beining Chen, Senior Lecturer, University of Sheffield

Andreas Kuglstatter, Research Scientist, Hoffmann-La Roche

Gisber Schneider, Professor, Goethe University

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Agenda Topics to include:

- Compound Management
- Chemogenomics
- Fragment Based Lead Detection
- Formulation

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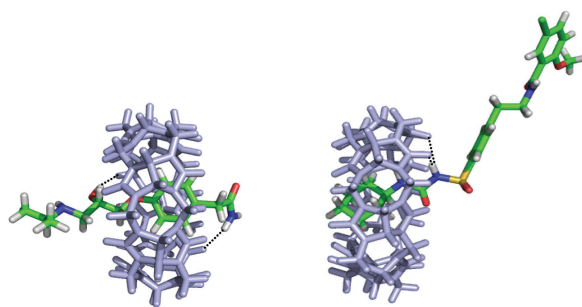
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Solid state stabilisation of the orally delivered drugs atenolol, glibenclamide, memantine and paracetamol through their complexation with cucurbit[7]uril

Fiona J. McInnes, Nahoum G. Anthony, Alan R. Kennedy and Nial J. Wheate*

The inclusion of the cardiovascular β -blocker drug atenolol, the antidiabetic drug glibenclamide, the Alzheimer's drug memantine and the analgesic paracetamol by cucurbit[7]uril has been studied by NMR, ESI-MS, molecular modelling, fluorescence displacement assays and DSC.

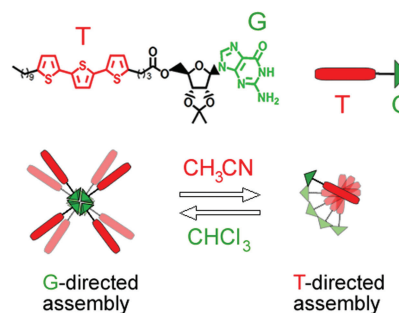


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Solvent-induced switching between two supramolecular assemblies of a guanosine–terthiophene conjugate

Silvia Pieraccini, Sara Bonacchi, Stefano Lena, Stefano Masiero, Marco Montalti, Nelsi Zaccheroni and Gian Piero Spada*

The guanosine–terthiophene conjugate undergoes a pronounced variation of its supramolecular organisation from a guanine-directed to a terthienyl-directed assembly by changing the polarity of the solvent.

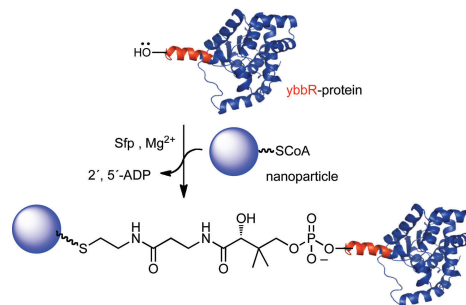


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Site-selective immobilisation of functional enzymes on to polystyrene nanoparticles

Lu Shin Wong,* Krzysztof Okrasa and Jason Micklefield*

Site-selective covalent immobilisation of ybbR tagged proteins, including functional enzymes, on to nanoparticles derivatised with CoA is achieved in a mild and efficient reaction catalysed by the promiscuous phosphopantetheinyl transferase Sfp.

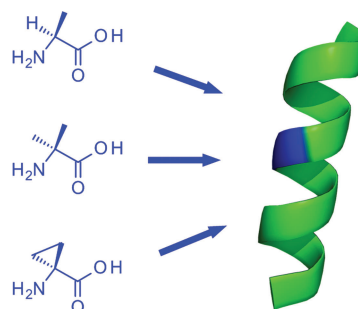


788

Helix propensities of conformationally restricted amino acids. Non-natural substitutes for helix breaking proline and helix forming alanine

Miriam Alías, Sara Ayuso-Tejedor, Juan Fernández-Recio, Carlos Cativiela* and Javier Sancho*

Helix propagation parameters of the conformationally restricted amino acids Aib and Ac3c as an aid for helical peptide designing.



2010 Meetings (in chronological order)

NF- κ B in Inflammation & Disease
 Advances in Biopharmaceuticals
 Structural Biology
 Structural Genomics: Expanding the Horizons of Structural Biology
 Triglycerides & Triglyceride-Rich Particles in Health & Disease (new!)
 Alzheimer's Disease Beyond A β
 Molecular Basis for Biological Membrane Organization & Dynamics
 HIV Biology & Pathogenesis
 RNA Silencing: Mechanism, Biology & Application
 Molecular Basis for Chromatin Structure & Regulation
 Hypoxia: Molecular Mechanisms of Oxygen Sensing & Response Pathways
 Adipose Tissue Biology
 Neuronal Control of Appetite, Metabolism & Weight*
 New Insights into Healthspan & Diseases of Aging
 Role of Inflammation in Oncogenesis
 Molecular and Cellular Biology of Immune Escape in Cancer
 Advances in Molecular Mechanisms of Atherosclerosis
 The Macrophage: Intersection of Pathogenic & Protective Inflammation
 Antibiotics & Resistance: Challenges & Solutions (new!)
 Stem Cell Differentiation & Dedifferentiation
 Cell Biology of Virus Entry, Replication & Pathogenesis
 RNA Silencing Mechanisms in Plants (new!)
 Tolerance & Autoimmunity
 Cilia, Signaling & Human Disease
 Lymphocyte Activation & Gene Expression
 Angiogenesis in Health & Disease
 Cardiovascular Development & Repair
 Biomolecular Interaction Networks: Function & Disease (new!)
 Cell Death Pathways: Apoptosis, Autophagy & Necrosis
 Metabolism & Cancer Progression (new!)
 Receptors and Signaling in Plant Development & Biotic Interactions
 HIV Vaccines
 Viral Immunity
 Nuclear Receptors: Signaling, Gene Regulation & Cancer
 Nuclear Receptors: Development, Physiology & Disease
 New Paradigms in Cancer Therapeutics
 Integration of Developmental Signaling Pathways
 G Protein-Coupled Receptors
 Dynamics of Eukaryotic Transcription During Development
 Synapses: Formation, Function & Misfunction
 Towards Defining the Pathophysiology of Autistic Behavior
 Malaria: New Approaches to Understanding Host-Parasite Interactions
 Molecular Targets for Control of Vector-Borne Diseases: Bridging Lab & Field Research*
 Islet Biology
 Diabetes
 Computer-Aided Drug Design
 New Directions in Small Molecule Drug Discovery (new!)
 Developmental Origins & Epigenesis in Human Health and Disease (new!)
 Bioactive Lipids: Biochemistry & Diseases
 Innate Immunity: Mechanisms Linking with Adaptive Immunity



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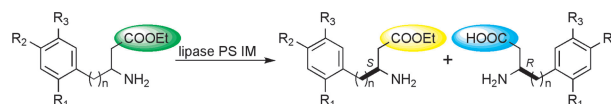
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Improved enzymatic syntheses of valuable β -arylalkyl- β -amino acid enantiomers

Gábor Tasnádi, Enikő Forró and Ferenc Fülöp*

The enantioselective ($E \sim 200$) *Burkholderia cepacia*-catalysed hydrolyses of β -amino esters with H_2O (0.5 equiv.) in *t*-BuOMe or in *i*-Pr₂O at 45 °C are described. The enantiomers were prepared with high enantiomeric excesses ($ee > 96\%$) and in good yields ($> 42\%$).

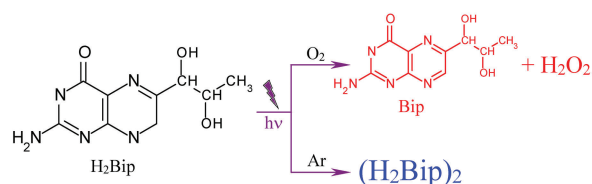


800

Photochemistry of dihydrobiopterin in aqueous solution

Mariana Vignoni, Franco M. Cabrerizo, Carolina Lorente, Catherine Claparols, Esther Oliveros* and Andrés H. Thomas*

Photooxidation of pterins takes place *in vivo* under pathological conditions. We have investigated the photochemistry of dihydrobiopterin and discuss the mechanisms involved.

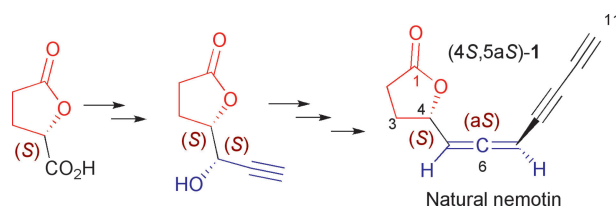


811

The enantioselective total synthesis of nemotin

Ya-Jun Jian and Yikang Wu*

The gross structure of nemotin was established in the 1950s, but the stereochemistry remains unknown to date. Now, with the aid of an enantioselective synthesis, the (4*S*,5*aS*) configuration among the four possible alternatives is shown to represent the natural product.

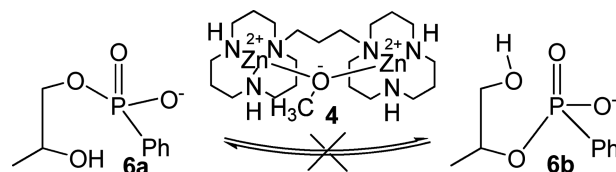


822

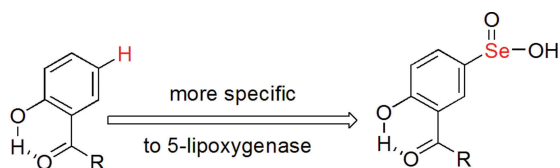
On the question of stepwise *vs.* concerted cleavage of RNA models promoted by a synthetic dinuclear Zn(II) complex in methanol: implementation of a noncleavable phosphonate probe

David R. Edwards, Wing-Yin Tsang, Alexei A. Neverov and R. Stan Brown*

$\text{Zn}(\text{II})_2$ complex (**4**) does not promote the interconversion of isomeric phosphonates (**6a**, **6b**), the only products of the reaction being 1,2-propanediol and *O*-methyl phenylphosphonate.



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R = acid, amide or ester

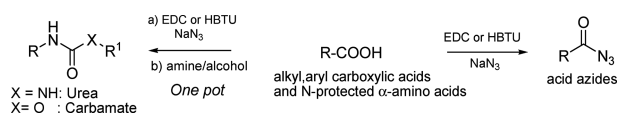
or its anhydride

5-Selenization of salicylic acid derivatives yielded isoform-specific 5-lipoxygenase inhibitors

Sun-Chol Yu, Hartmut Kuhn,* Constantin-Gabriel Daniliuc, Igor Ivanov, Peter G. Jones and Wolf-Walther du Mont*

5-Seleninic acids or anhydrides of salicylic acid amides or esters are promising inhibitors more selective to recombinant human 5-lipoxygenase than to rabbit reticulocyte 12/15-lipoxygenase.

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X = NH: Urea

X = O : Carbamate

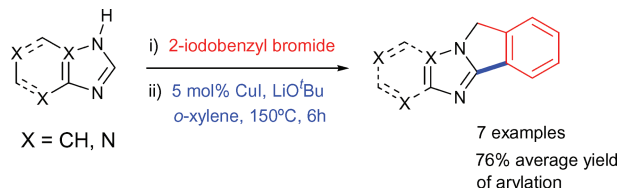
alkyl, aryl carboxylic acids and N-protected α -amino acids

New and simple synthesis of acid azides, ureas and carbamates from carboxylic acids: application of peptide coupling agents EDC and HBTU

Vommina V. Sureshbabu,* H. S. Lalithamba, N. Narendra and H. P. Hemantha

Acid azides have been efficiently prepared from carboxylic acids using peptide coupling agents EDC or HBTU; and the reaction has been extended to the one pot synthesis of ureas and carbamates from carboxylic acids

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X = CH, N

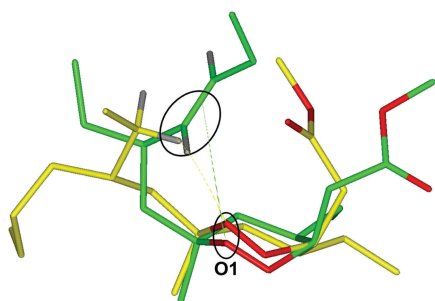
7 examples
76% average yield of arylation

Ligand-free copper(I)-catalysed intramolecular direct C–H functionalization of azoles

Nekane Barbero, Raul SanMartin* and Esther Domínguez*

The first examples of copper-catalysed intramolecular direct C-arylation of azaheterocycles for the synthesis of complex heterofused compounds is presented, featuring an unprecedented arylation *via* C–H activation of 9*H*-purine and 4-azabenzimidazole.

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Insight into the mechanism of action of plakortins, simple 1,2-dioxane antimalarials

Orazio Tagliatela-Scafati, Ernesto Fattorusso, Adriana Romano, Fernando Scala, Vincenzo Barone, Paola Cimino, Emiliano Stendardo, Bruno Catalanotti, Marco Persico and Caterina Fattorusso*

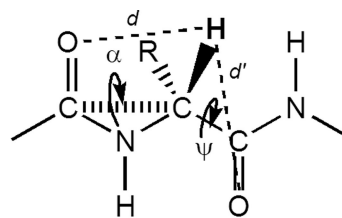
Computational calculations and chemical reactions give insight into the mechanism of the antimalarial action of plakortin and dihydroplakortin, simple 1,2-dioxanes isolated from a marine sponge.

857

A new model for mapping the peptide backbone: predicting proton chemical shifts in proteins

José Luis Barneto,* Martín Avalos, Reyes Babiano, Pedro Cintas, José Luis Jiménez and Juan Carlos Palacios

This study provides a methodology to correlate empirical chemical shifts (at the alpha-proton) in proteins with geometrical data (distances and dihedral angles) calculated at the B3PW91/6-31G* level.



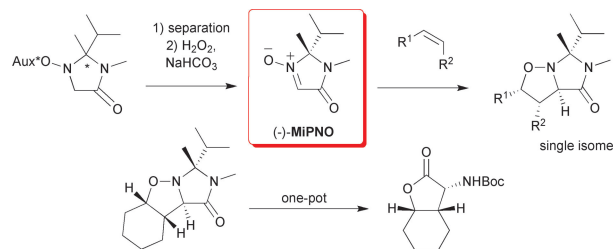
$$\delta = 2.87 + \sigma_R + 1.32 \cos^2 \frac{(\alpha - 25)}{d} + 0.75 \cos^2 \frac{(\psi - 10)/2}{d'}$$

864

MiPNO, a new chiral cyclic nitron for enantioselective amino acid synthesis: the cycloaddition approach

Maryse Thiverny, Christian Philouze, Pierre Yves Chavant and Véronique Blandin*

Totally regio- and diastereo-selective 1,3-dipolar cycloaddition reaction of various alkenes with MiPNO, a new chiral cyclic nitron, provides an expeditious enantioselective access to unusual γ -hydroxy α -amino acids.

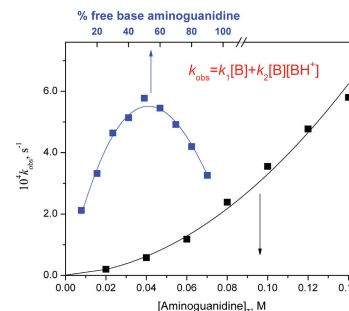


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Mechanism of general acid–base catalysis in transesterification of an RNA model phosphodiester studied with strongly basic catalysts

David O. Corona-Martínez, Olga Taran and Anatoly K. Yatsimirsky*

Classical “bell-shaped” second-order kinetics of general acid–base catalysis in transesterification of an RNA model substrate absent in aqueous buffers is observed in guanidine and amidine buffers in 80% vol aqueous DMSO.

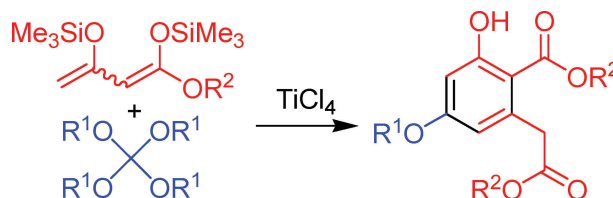


881

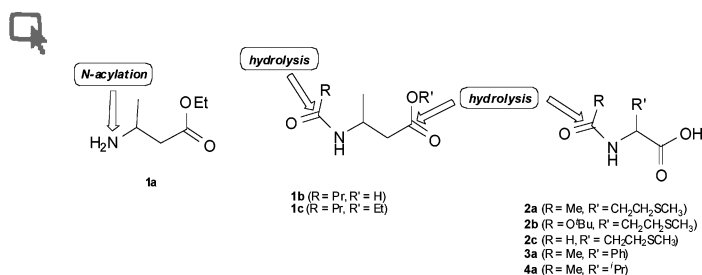
Synthesis of 3-hydroxy-5-alkoxyhomophthalates by domino ‘2 : 1-coupling/intramolecular aldol condensation’ reactions of 1,3-bis(trimethylsilyloxy)-1,3-butadienes with tetraalkoxymethanes

Mathias Lubbe and Peter Langer*

The first domino ‘2 : 1 condensation/intramolecular aldol’ reactions of 1,3-bis(trimethylsilyloxy)-1,3-butadiene with tetraalkoxymethanes provide a convenient approach to 3-hydroxy-5-alkoxyhomophthalates.



886

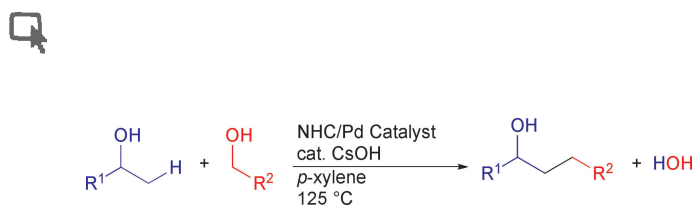


Formation and hydrolysis of amide bonds by lipase A from *Candida antarctica*; exceptional features

Arto Liljeblad,* Pauli Kallio, Marita Vainio, Jarmo Niemi and Liisa T. Kanerva

Exceptional lipase: the ability of lipase A from *Candida antarctica* (CAL-A) to form and hydrolyze amide bonds was studied with **1a–c**, **2a–c**, **3a** and **4a**. The possible role of enzyme contaminants in the reactions was studied by fractionation and sequence-based identification of the commercial CAL-A preparation Cat#ICR-112 (Codexis).

896

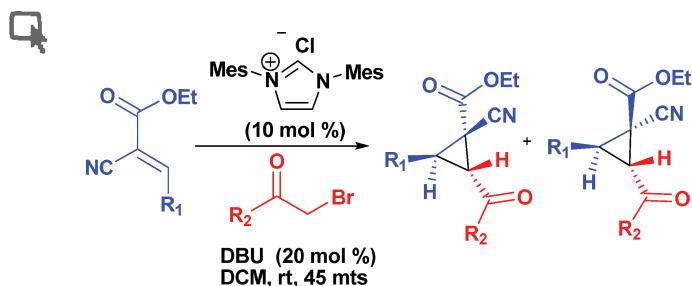


Cross-coupling reaction of alcohols for carbon–carbon bond formation using pincer-type NHC/palladium catalysts

Osamu Kose and Susumu Saito*

A cross-coupling reaction of different alcohols was achieved using a pincer-type NHC/PdBr complex as the catalyst precursor, and the reaction, under either Ar or H₂ gas, displayed a broad substrate scope with respect to both primary and secondary alcohol components, with high product alcohol selectivity.

901

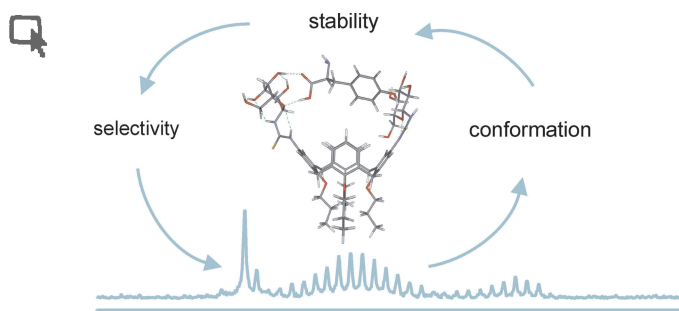


Nucleophilic heterocyclic carbene as a novel catalyst for cyclopropanation of cyano acrylates

Anabha E. Raveendran, Rony Rajan Paul, Eringathodi Suresh and Vijay Nair*

Nucleophilic heterocyclic carbenes (NHCs) have been used for the first time as catalysts in the cyclopropanation of ethyl cyanocinnamates with phenacyl bromide by Michael-initiated ring-closure (MIRC).


906



Glucosylthioureidocalix[4]arenes: Synthesis, conformations and gas phase recognition of amino acids

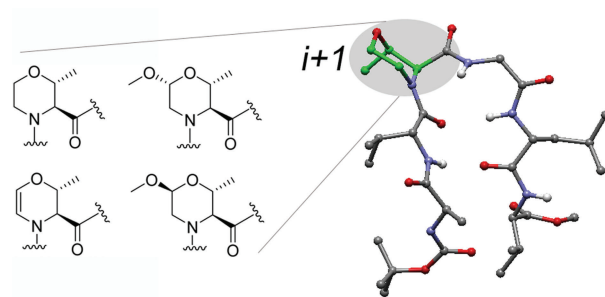
Mika Torvinen, Raisa Neitola, Francesco Sansone, Laura Baldini, Rocco Ungaro, Alessandro Casnati,* Pirjo Vainiotalo* and Elina Kalenius*

Conformational properties of glucosylcalixarenes and their selective complexation with amino acids have been clarified in solution and gas-phase.

 **Evaluation of stereochemically dense morpholine-based scaffolds as proline surrogates in β -turn peptides**

Filippo Sladojevich, Antonio Guarna and Andrea Trabocchi*

NMR analysis of stereochemically dense morpholine-based scaffolds as proline surrogates in β -turn peptides revealed an adaptive behaviour in generating turn conformations stabilized by intramolecular hydrogen-bonds with no significant loss of the secondary framework.



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