Organic & Biomolecular Chemistry

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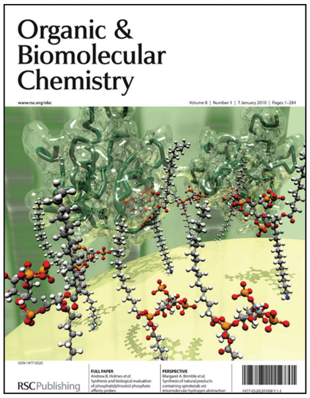
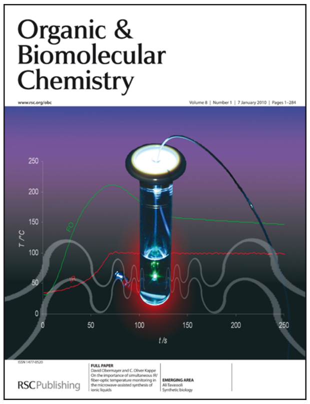
IN THIS ISSUE

ISSN 1477-0520 CODEN OBCRAK 8(1) 1–284 (2010)



Cover
See Andrew B. Holmes *et al.*, pp. 66–76.
The cover page illustrates a phosphatidylinositol (3,4,5)-trisphosphate analogue bound to an affinity bead interacting with the PH domain of GRP1, representing the pulldown of intracellular signalling proteins from the cytosolic extracts of pig neutrophil and colon cancer cells (Created by Karl Harrison, University of Oxford).

Image reproduced by permission of Andrew B. Holmes from *Org. Biomol. Chem.*, 2010, **8**, 66.



Inside cover
See David Obermayer and C. Oliver Kappe, pp. 114–121.
Seeing is believing: infrared sensors used in most of today's microwave reactors can easily lead to incorrect reaction temperature measurements – internal fiber-optic probes do a much better job.

Image reproduced by permission of C. Oliver Kappe from *Org. Biomol. Chem.*, 2010, **8**, 114.

EDITORIAL

21

Warmest wishes to all *Organic & Biomolecular Chemistry* authors, referees and readers for 2010

Following the success of the last six years, 2009 was another superb year for *Organic & Biomolecular Chemistry*.

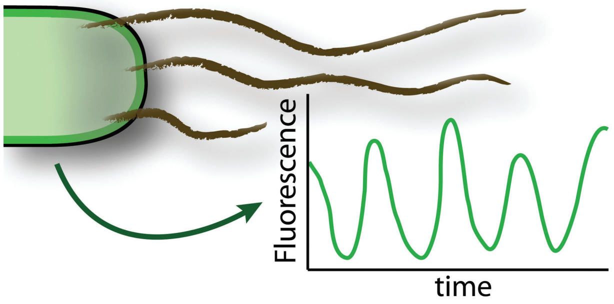
EMERGING AREA

24

Synthetic biology

Ali Tavassoli*

There is currently much excitement surrounding the rapidly growing discipline of synthetic biology, which utilizes the design and construction principles of engineering to develop, evolve and standardize biological components and systems. Here we discuss the achievements of the field so far, and go on to outline its potential future directions.



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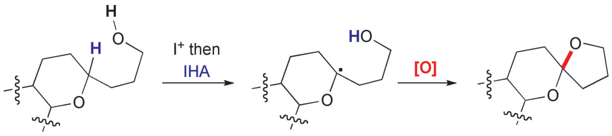
PERSPECTIVE

29

Synthesis of natural products containing spiroketals *via* intramolecular hydrogen abstraction

Jonathan Sperry, Yen-Cheng (William) Liu and
Margaret A. Brimble*

Although known for over a quarter of a century, the oxidative radical cyclisation approach to spiroketals has found limited use in natural product synthesis in comparison to classical approaches. Its successful application in this field of research forms the subject of this perspective.



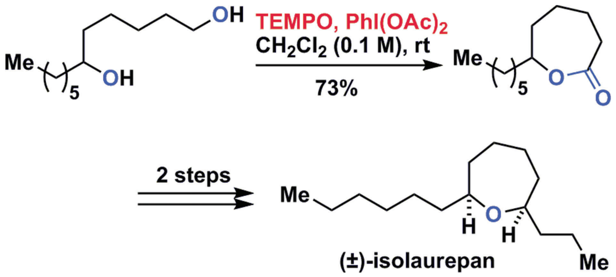
COMMUNICATIONS

39

Highly efficient synthesis of medium-sized lactones *via* oxidative lactonization: concise total synthesis of isolaurepan

Makoto Ebine, Yuto Suga, Haruhiko Fuwa* and
Makoto Sasaki

Oxidative lactonization of 1,6- and 1,7-diols under the influence of a catalytic amount of TEMPO in the presence of PhI(OAc)$_2$ afforded seven- and eight-membered lactones, respectively, in good yields. A concise total synthesis of (±)-isolaurepan has been achieved based on this methodology.

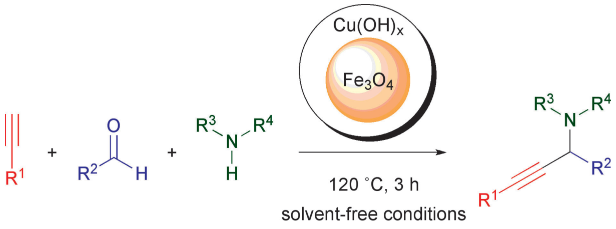


43

Impregnated copper on magnetite: an efficient and green catalyst for the multicomponent preparation of propargylamines under solvent free conditions

María J. Aliaga, Diego J. Ramón* and Miguel Yus

Impregnated copper on magnetite is an efficient and green catalyst for the multicomponent acetylene-Mannich reaction.

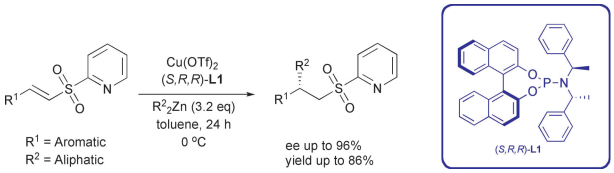


47

Catalytic asymmetric conjugate addition of dialkylzinc reagents to α,β-unsaturated sulfones

Pieter H. Bos, Beatriz Maciá, M. Ángeles Fernández-Ibáñez,
Adriaan J. Minnaard and Ben L. Feringa*

An efficient method is reported for the highly enantioselective copper-catalyzed conjugate addition of dialkylzinc reagents to α,β-unsaturated sulfones using a monodentate phosphoramidite ligand.



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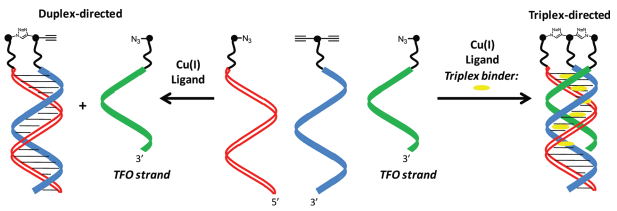
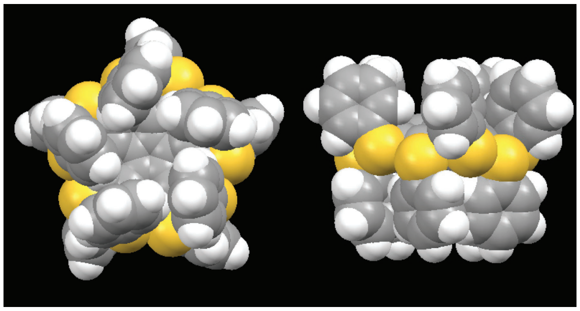


Small molecule induced control in duplex and triplex DNA-directed chemical reactions

Mikkel F. Jacobsen, Jens B. Ravnsbæk and Kurt V. Gothelf*

Triplex DNA binders can effectively control copper-catalysed alkyne–azide click reactions in DNA architecture, such that either duplex or triplex DNA directed reactions of terminally attached azides and alkynes occur, in the absence or presence of triplex DNA binder, respectively.

53

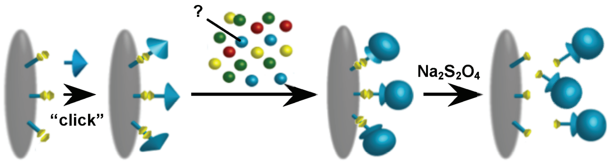


Synthesis, structure and properties of decakis(phenylthio)corannulene

Kim K. Baldridge,* Kenneth I. Hardcastle, T. Jon Seiders and Jay S. Siegel*

Decakis(phenylthio)corannulene adopts a pinwheel conformation with multiple T-shaped (edge-to-face) arene interactions.

56

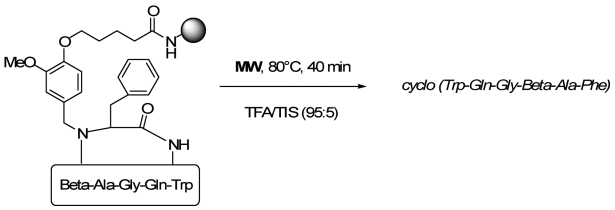


Synthesis and application of a new cleavable linker for "click"-based affinity chromatography

Felicetta Landi, Conny M. Johansson,
Dominic J. Campopiano and Alison N. Hulme*

"Click" coupling of a ligand to an azo-functionalised linker allows affinity capture and separation of proteins from a complex mixture (such as fetal bovine serum) and mild release with sodium dithionite.

60



Development of a new microwave-assisted cleavable backbone amide linker (BAL): a comparative study

Stijn Claerhout, Thibault Duchène, Dirk Tourwé and Erik V. Van der Eycken*

A thorough comparative study to demonstrate the properties of a new microwave labile backbone amide linker is presented.

DALTON DIVISION AND ORGANIC DIVISION

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

13 - 15 September 2010
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Keynote speakers

Keith Fagnou
University of Ottawa, Canada

Jennifer Love
The University of British Columbia, Canada

William D. Jones
University of Rochester, USA

Aiwen Lei
Wuhan University, China

Invited speakers

Robin Bedford
University of Bristol, UK

John M. Brown
University of Oxford, UK

Stuart Macgregor
Heriot-Watt University, Edinburgh, UK

Hans de Vries
DSM Pharmaceutical Products, The Netherlands

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Registration will open in spring 2010.

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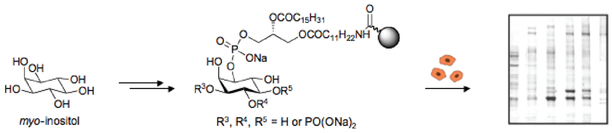
66

Synthesis and biological evaluation of phosphatidylinositol phosphate affinity probes

S. J. Conway, J. Gardiner,* S. J. A. Grove, M. K. Johns, Z.-Y. Lim, G. F. Painter, D. E. J. E. Robinson,* C. Schieber, J. W. Thuring, L. S.-M. Wong, M.-X. Yin, A. W. Burgess, B. Catimel, P. T. Hawkins, N. T. Ktistakis, L. R. Stephens and A. B. Holmes*

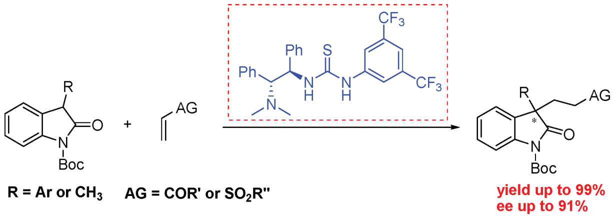


A versatile approach to the synthesis of the complete family of phosphatidylinositol phosphate (PIP) analogues and their use as affinity probes are described.

77

Asymmetric Michael addition reaction of 3-substituted-*N*-Boc oxindoles to activated terminal alkenes catalyzed by a bifunctional tertiary-amine thiourea catalyst

Xin Li, Zhi-Guo Xi, Sanzhong Luo* and Jin-Pei Cheng*

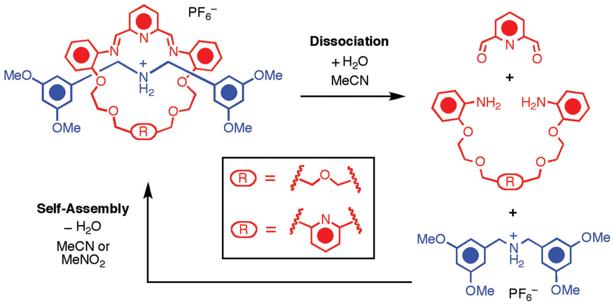


An enantioselective Michael addition reaction of 3-substituted oxindoles to terminal alkenes which realized the construction of an all carbon-substituted quaternary stereocenter has been reported using catalysis by a bifunctional tertiary-amine thiourea organocatalyst.

83

The stability of imine-containing dynamic [2]rotaxanes to hydrolysis

Ken Cham-Fai Leung,* Wing-Yan Wong, Fabio Aricó, Philip C. Haussmann and J. Fraser Stoddart*

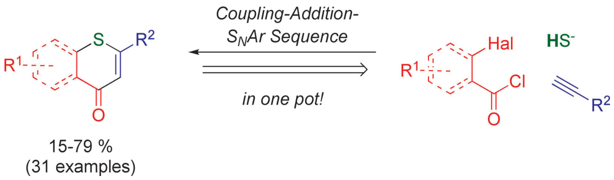


The self-assembly as well as the dissociation of two dynamic [2]rotaxanes have been demonstrated. Somewhat surprisingly, the [2]rotaxanes show remarkable relative stabilities in the presence of large amounts of water. In particular, the dissociation rates of the rotaxanes toward hydrolysis have been determined.

90

Microwave-assisted three-component coupling-addition-S_NAr (CASNAR) sequences to annelated 4*H*-thiopyran-4-ones

Benjamin Willy, Walter Frank and Thomas J. J. Müller*



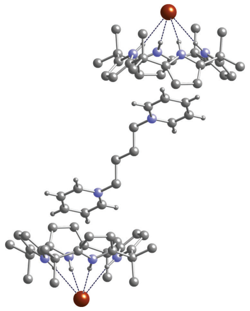
Annelated 4*H*-thiopyran-4-ones can be readily synthesized from readily available (het)aroyl chlorides, alkynes and sodium sulfide nonahydrate in good yields by a microwave-assisted consecutive one-pot three-component reaction.

96

Bis-cation salt complexation by *meso*-octamethylcalix[4]pyrrole: linking complexes in solution and in the solid state

Claudia Caltagirone, Nathan L. Bill, Dustin E. Gross, Mark E. Light, Jonathan L. Sessler* and Philip A. Gale*

Bis-pyridinium and bis-imidazolium cations can be used to link calix[4]pyrrole anion complexes in solution and the solid state as the first step to producing ordered arrays of calixpyrroles for use in new materials applications.

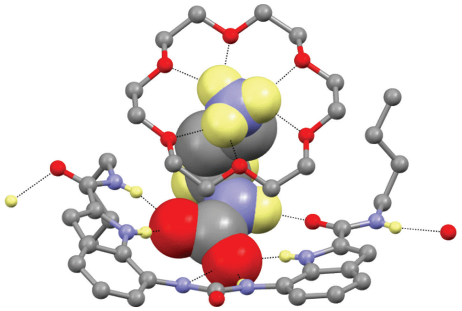


100

Carbamate complexation by urea-based receptors: studies in solution and the solid state

Peter R. Edwards, Jennifer R. Hiscock, Philip A. Gale* and Mark E. Light

CO_2 can be captured by amines or tetrahydropyrimidine to form carbamates which can then be stabilised by complexation with receptors containing multiple hydrogen bond donors.

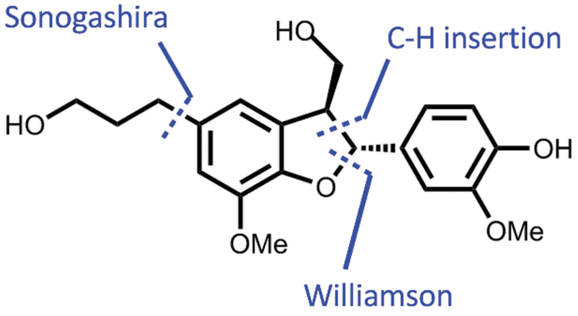


107

Synthesis of dihydrodehydrodiconiferyl alcohol: the revised structure of lawsonicin

Junxiu Meng, Tao Jiang, Huma Aslam Bhatti, Bina S. Siddiqui, Sally Dixon* and Jeremy D. Kilburn*

Synthesis of dihydrobenzo[*b*]furan neolignan, dihydrodehydrodiconiferyl alcohol, *via* a concise route involving $Rh_2[S\text{-DOSP}]_4$-catalysed intramolecular C–H insertion, is reported. The *rac*-2,3-*trans*-epimer is found to have identical spectral data to the earlier reported natural product, lawsonicin, whose structure is revised.

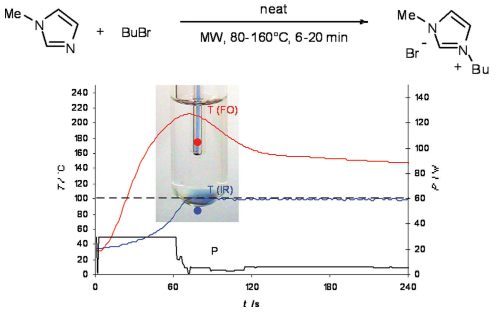


114

On the importance of simultaneous infrared/fiber-optic temperature monitoring in the microwave-assisted synthesis of ionic liquids

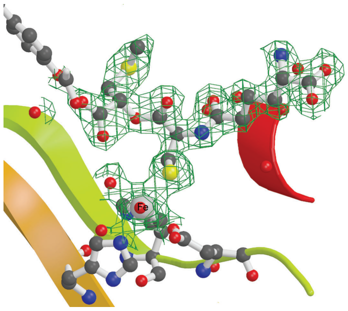
David Obermayer and C. Oliver Kappe*

Infrared sensors give erroneous temperature readings and can not be used for microwave chemistry involving ionic liquids.



PAPERS

122

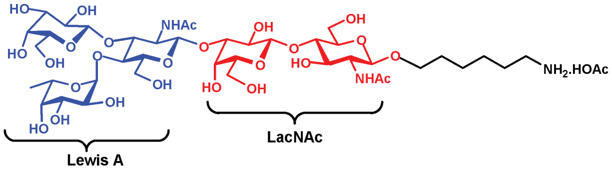


The crystal structure of an LLL-configured depsipeptide substrate analogue bound to isopenicillin N synthase

Wei Ge, Ian J. Clifton, Jeanette E. Stok,
Robert M. Adlington, Jack E. Baldwin* and
Peter J. Rutledge*

Stereochemistry is all-important to enzyme catalysis, so what will isopenicillin N synthase, a key player in penicillin biosynthesis, make of an LLL-configured depsipeptide substrate analogue when its natural substrate is an LLD-configured tripeptide?

128

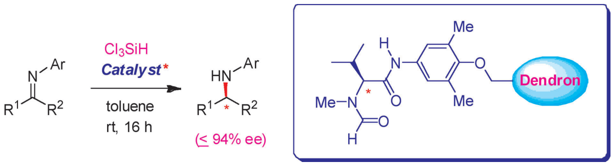


Total synthesis of Le^A-LacNAc pentasaccharide as a ligand for *Clostridium difficile* toxin A

Ping Zhang, Kenneth Ng and Chang-Chun Ling*

Le^A-LacNAc was found to be a ligand for toxin A released by *Clostridium difficile.* Here we report the efficient synthesis of the pentasaccharide for use to characterize the carbohydrate-binding site of the toxin by X-ray crystallography.

137

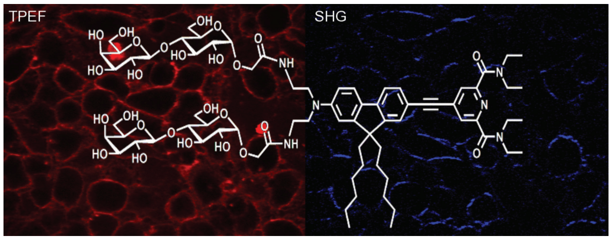


Dendron-anchored organocatalysts: the asymmetric reduction of imines with trichlorosilane, catalysed by an amino acid-derived formamide appended to a dendron

Marek Figlus, Stuart T. Caldwell, Dawid Walas,
Gulen Yesilbag, Graeme Cooke,* Pavel Kočovský,*
Andrei V. Malkov* and Amitav Sanyal*

Ketimines **1a–f** can be reduced with Cl_3SiH in the presence of the Lewis-basic *N*-methylvaline-derived formamide catalyst (5 mol%) anchored to a soluble dendron (**11c**) with ≤94% ee; this protocol represents a simplification of the isolation procedure and recovery of the catalyst.

142

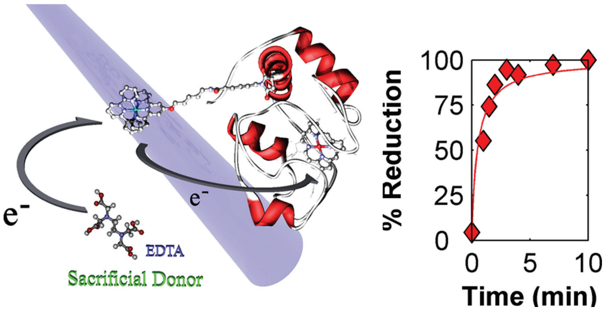


Neutral push-pull chromophores for nonlinear optical imaging of cell membranes

Cyril Barsu, Rouba Cheaib, Stéphane Chambert,
Yves Queneau, Olivier Maury, Davy Cottet, Hartmut Wege,
Julien Douady, Yann Bretonnière* and Chantal Andraud*

A series of amphiphilic second order nonlinear probes for cell membrane imaging was studied by combined two-photon excited fluorescence (TPEF) and second harmonic generation (SHG) microscopies.

151

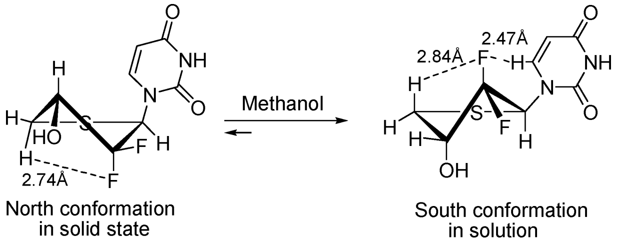


Synthesis and room temperature photo-induced electron transfer in biologically active bis(terpyridine)ruthenium(II)–cytochrome *c* bioconjugates and the effect of solvents on the bioconjugation of cytochrome *c*

Joshua R. Peterson, Trevor A. Smith and Pall Thordarson*

Room temperature photo-induced electron transfer studies of bis(terpyridine)ruthenium(II) cytochrome *c* bioconjugates show that the protein is effectively reduced within 5 min in the presence of 20 mM EDTA.

163

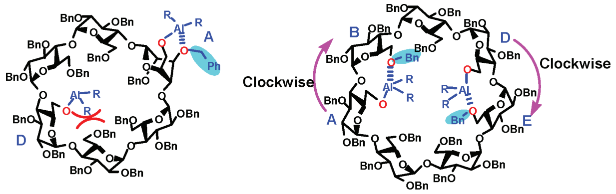


Synthesis and conformational analysis of D-2′-deoxy-2′,2′-difluoro-4′-dihydro-4′-thionucleosides

Feng Zheng, Lin Fu, Renxiao Wang and Feng-Ling Qing*

An efficient synthesis of D-2′-deoxy-2′,2′-difluoro-4′-thionucleosides is described. The conformations of D-2′-deoxy-2′,2′-difluoro-4′-thiouridine were studied by X-ray crystallography, NMR spectroscopy and molecular modeling in an attempt to explore the roles of the two *gem*-difluorine atoms in the conformer preferences of the thionucleosides.

171

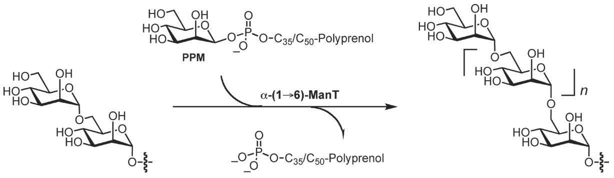


DIBAL-H mediated triple and quadruple debenzylations of perbenzylated cyclodextrins

Girish K. Rawal, Shikha Rani, Sandra Ward and Chang-Chun Ling*

Sequential triple and quadruple debenzylations by diisobutylaluminium hydride were investigated for α-, β- and γ-cyclodextrins. A distinct regioselective debenzylation path was observed for α-CD compared to β- and γ-cyclodextrins.

181



Epimeric and amino disaccharide analogs as probes of an α-(1→6)-mannosyltransferase involved in mycobacterial lipoarabinomannan biosynthesis

Pui Hang Tam and Todd L. Lowary

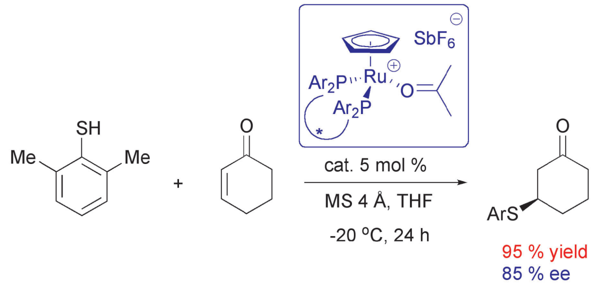
A panel of disaccharide analogs have been synthesized and used to probe a mannosyltransferase involved in lipoarabinomannan biosynthesis in mycobacteria, including the human pathogen *Mycobacterium tuberculosis*.

193

Asymmetric ruthenium-catalyzed 1,4-additions of aryl thiols to enones

Andrei Bădoiu, Gerald Bernardinelli, Céline Besnard and E. Peter Kündig*

Ruthenium half-sandwich Lewis-acids can efficiently catalyze 1,4-additions of aryl thiols to enones with remarkable levels of activity and selectivity, in spite of stereocontrol complexity and potential catalyst inhibition. NMR experiments provide an insight to the intricate equilibria governing the reaction mechanism.

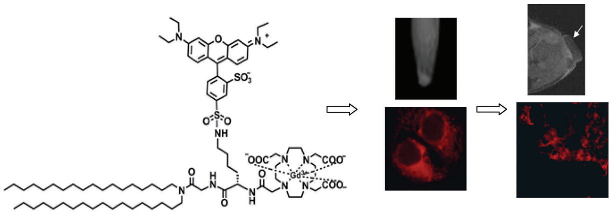


201

A novel bimodal lipidic contrast agent for cellular labelling and tumour MRI

Nazila Kamaly, Tammy Kalber, Gavin Kenny, Jimmy Bell, Michael Jorgensen* and Andrew Miller*

Gd·DOTA·Rhoda·DSA: a novel bimodal lipidic MRI contrast agent.

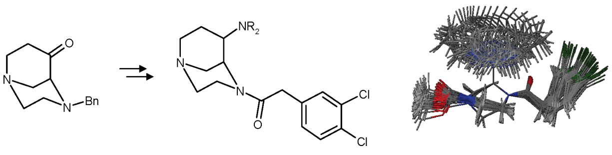


212

Stereoselective synthesis and structure–affinity relationships of bicyclic κ receptor agonists

Daniel Kracht, Elisabeth Rack, Dirk Schepmann, Roland Fröhlich and Bernhard Wünsch*

The bioactive conformation of flexible κ agonists was approached by the design, synthesis and pharmacological evaluation of conformationally constrained 1,4-diazabicyclo[3.3.1]nonanes. It was shown that *endo*-orientation of the amino group is crucial for high κ receptor affinity.

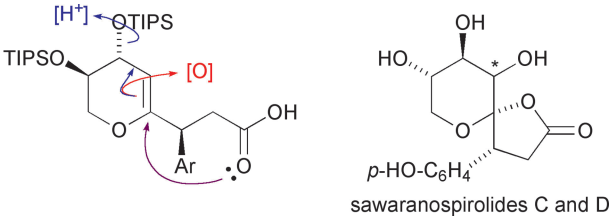


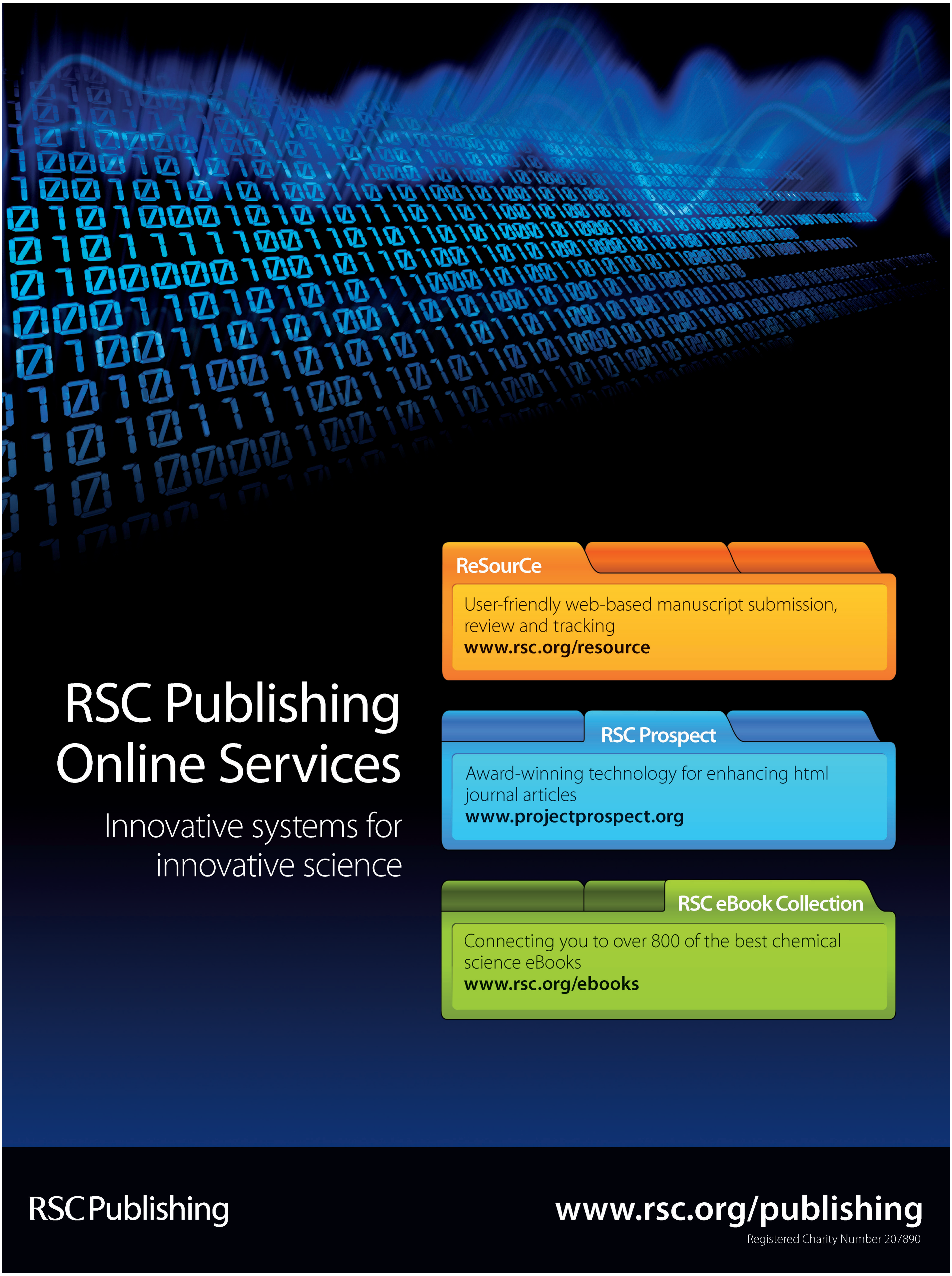
226

Oxidative spirocyclisation routes towards the sawaranospirolides. Synthesis of *ent*-sawaranospirolides C and D

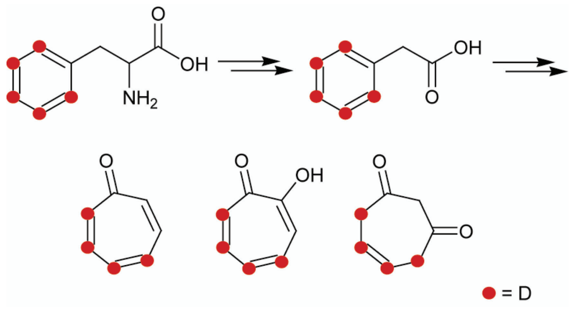
Jeremy Robertson,* Praful T. Chovatia, Thomas G. Fowler, Jonathan M. Withey and Daniel J. Woollaston

ent-Sawaranospirolides C and D are prepared by the electrophilic spirocyclisation of a 3-(dihydropyran-2-yl)propanoic acid derivative.





234

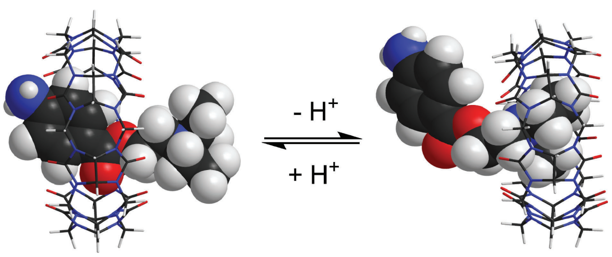


Identification and biosynthesis of tropone derivatives and sulfur volatiles produced by bacteria of the marine *Roseobacter* clade

Verena Thiel, Thorsten Brinkhoff, Jeroen S. Dickschat, Susanne Wickel, Jörg Grunenberg, Irene Wagner-Döbler, Meinhard Simon and Stefan Schulz*

The volatile tropone, its derivatives, and several thioesters are produced by *Phaeobacter gallaeciensis*, a bacterium of the *Roseobacter* clade that dominates many marine bacterial communities.

247

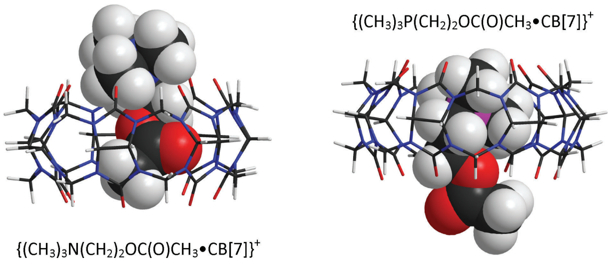


Host–guest complexations of local anaesthetics by cucurbit[7]uril in aqueous solution

Ian W. Wyman and Donal H. Macartney*

The cucurbit[7]uril host molecule binds to procaine, and other local anaesthetic drugs, 2–3 orders of magnitude stronger than β-cyclodextrin in aqueous solution, resulting in increases in the value of pK_1 and enhanced fluorescence.

253

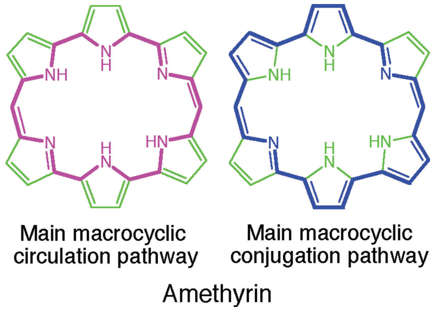


Cucurbit[7]uril host–guest complexes of cholines and phosphonium cholines in aqueous solution

Ian W. Wyman and Donal H. Macartney*

The cucurbit[7]uril host molecule forms host–guest complexes with a series of cholines and phosphonium cholines ($R_3XCH_2CHR'OR''^+$) in aqueous solution, with stability constants and guest positioning that reflects the natures of the peralkylonium head groups (R_3X^+) and substituents (R′ and R″).

261



Prediction of the main macrocyclic conjugation pathway for porphyrinoids from the ring current distribution

Jun-ichi Aihara* and Masakazu Makino

Using our analytical theory of ring-current diamagnetism, main macrocyclic conjugation pathways in porphyrinoids were successfully predicted from the global ring current distributions.

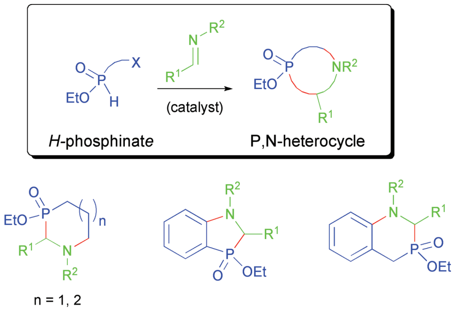
PAPERS

267

Facile P,N-heterocycle synthesis *via* tandem aminomethylation–cyclization of *H*-phosphinate building blocks

Clémence Queffélec and Jean-Luc Montchamp*

Various heterocycles containing phosphorus and nitrogen are easily synthesized from readily available *H*-phosphinate building blocks *via* aminomethylation and *in situ* cyclization through substitution or cross-coupling.

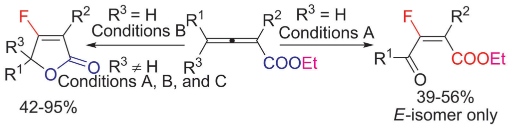


274

Study on the selectivity in the electrophilic monofluorination of 2,3-allenoates with Selectfluor^{TM}: an efficient synthesis of 4-fluoro-2(5*H*)-furanones and 3-fluoro-4-oxo-2(*E*)-alkenoates

Bo Lü, Chunling Fu and Shengming Ma*

4-Fluoro-2(5*H*)-furanones and (*E*)-3-fluoro-4-oxo-2-alkenoates were highly selectively generated from 2,4-disubstituted 2,3-allenoates with Selectfluor^{TM} in moderate to high yields under different reaction conditions. A mechanism has been proposed.



Conditions A: 3.0 equiv Selectfluor, 0.5 equiv H_2O, MeCN, 80 °C. Conditions B: 1.7-2.5 equiv Selectfluor, MeCN/H_2O = 2/1, 80 °C. Conditions C: 1.2 equiv Selectfluor, MeCN or $MeNO_2$, 80 °C.

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