

Organic & Biomolecular Chemistry

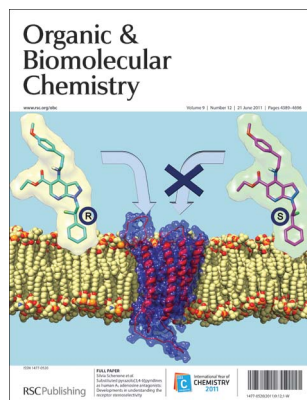
An international journal of synthetic, physical and biomolecular organic chemistry

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IN THIS ISSUE

ISSN 1477-0520 CODEN OBCRAK 9(12) 4389–4696 (2011)



Cover

See Tiziano Tuccinardi *et al.*, pp. 4448–4455.

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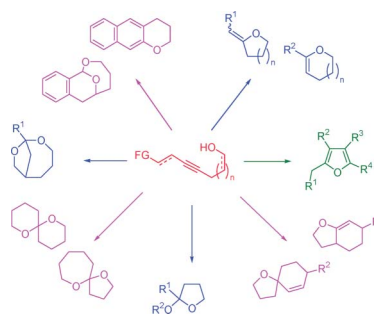
PERSPECTIVE

4405

Gold catalyzed oxycyclizations of alkynols and alkyndiols

Benito Alcaide,* Pedro Almendros* and José Miguel Alonso

Alkynols and alkyndiols represent excellent building blocks for gold-catalyzed oxycyclizations, leading to a large number of different cyclic structures in one single step.



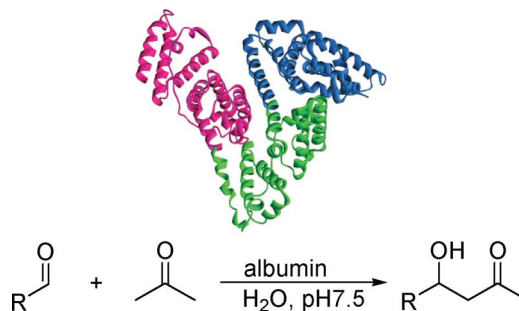
COMMUNICATIONS

4417

Aldolase activity of serum albumins

Fabio Benedetti, Federico Berti* and Silvia Bidoggia

Albumin does it. Bovine and human serum albumins catalyze the aldol reaction of aromatic aldehydes and acetone.



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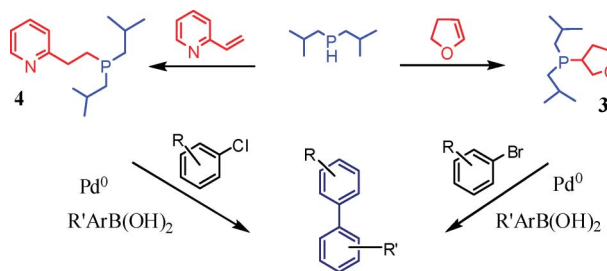
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4421

One step entry to P,O- and P,N-type heterocyclic tertiary phosphine ligands and application in Suzuki–Miyaura cross-coupling reactions

Ehsan Ullah, James McNulty,* Christine Kennedy and Al Robertson

Free-radical addition of diisobutylphosphine onto vinyl-heterocycles provides a one-pot entry to useful P-heterocyclic ligands **3** and **4**.

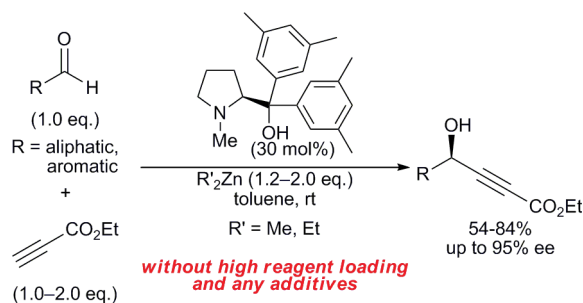


4425

Asymmetric alkylation of aldehydes with propiolates without high reagent loading and any additives

Naoto Kojima,* Shogo Nishijima, Kaoru Tsuge and Tetsuaki Tanaka*

The enantioselective addition of propiolates to aldehydes in the presence of a novel prolinol catalyst without high reagent loading and any additives was developed.

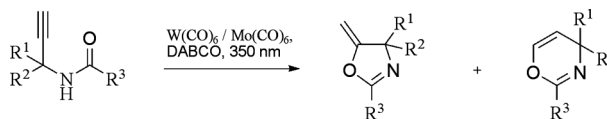


4429

Tungsten and molybdenum catalyst-mediated cyclisation of *N*-propargyl amides

Xiangjian Meng and Sunggak Kim*

W and Mo catalysts are very effective for the cyclisation of *N*-propargylic amides to afford the corresponding oxazolines or oxazines via *5-exo-dig* or *6-endo-dig* mode.

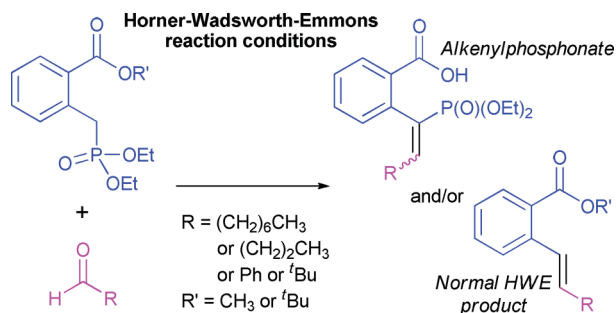


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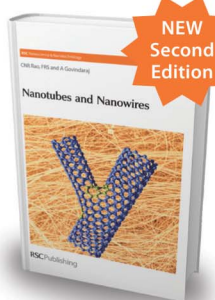
Alkenylphosphonates: unexpected products from reactions of methyl 2-[(diethoxyphosphoryl)methyl]benzoate under Horner–Wadsworth–Emmons conditions

Lynton J. Baird, Cédric Colombar, Claire Turner, Paul H. Teesdale-Spittle and Joanne E. Harvey*

Alkenylphosphonates are the major products from reactions of a 2-(methoxycarbonyl)benzylphosphonate with aldehydes under Horner–Wadsworth–Emmons reaction conditions.



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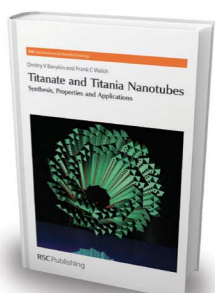
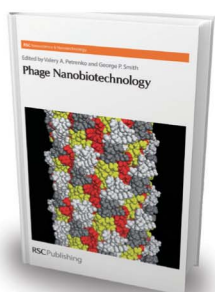
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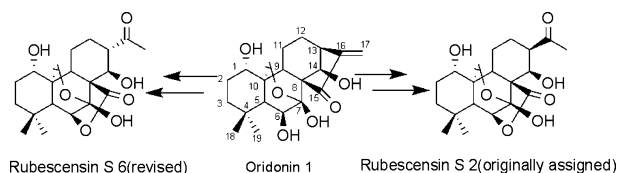
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4436

Synthesis and revision of stereochemistry of rubescensin S

Mei Zhang, Yangming Zhang, Wei Lu* and Fa-Jun Nan*

An effective two step transformation of oridonin to 15,16-seco-ent-kaurane skeleton is reported. We also achieved the conversion of one intermediate to natural product rubescensin S and revised its structure as a *13S* configuration although *13R* is reported in the literature.

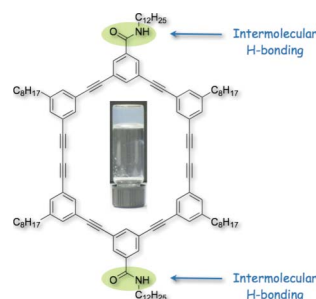


4440

H-Bonding-driven gel formation of a phenylacetylene macrocycle

Katy Cantin, Simon Rondeau-Gagné, Jules Roméo Néabo, Maxime Daigle and Jean-Francois Morin*

An amide-containing phenylacetylene macrocycle (PAM) has been synthesized and its H-bonding-driven gelation properties were studied in different solvents.

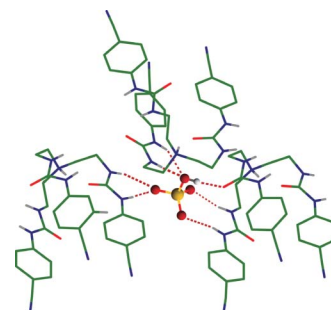


4444

Seven-coordinate anion complex with a tren-based urea: Binding discrepancy of hydrogen sulfate in solid and solution states

Avijit Pramanik, Bethrice Thompson, Trina Hayes, Kimberly Tucker, Douglas R. Powell, Peter V. Bonnesen, Erick D. Ellis, Ken S. Lee, Hongtao Yu and Md. Alamgir Hossain*

Structural characterization of a hydrogen sulfate complex with tren-based urea suggests that the anion is heptacoordinated with six $\text{NH} \cdots \text{O}$ bonds and one $\text{OH} \cdots \text{O}$ bond.



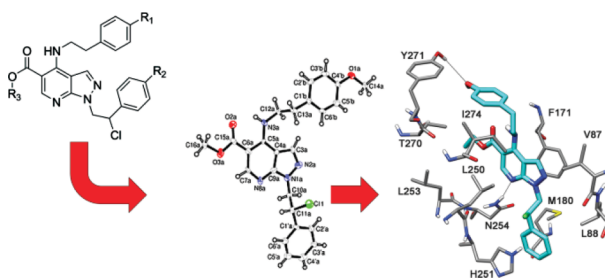
PAPERS

4448

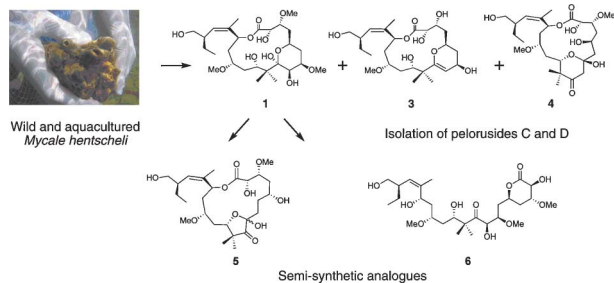
Substituted pyrazolo[3,4-*b*]pyridines as human A₁ adenosine antagonists: Developments in understanding the receptor stereoselectivity

Tiziano Tuccinardi, Alessandra Tania Zizzari, Chiara Brullo, Simona Daniele, Francesca Musumeci, Silvia Schenone,* Maria Letizia Trincavelli, Claudia Martini, Adriano Martinelli, Gianluca Giorgi and Maurizio Botta

This is one of the first attempts to design highly potent A₁AR antagonists taking into consideration the compound's chirality.



4456

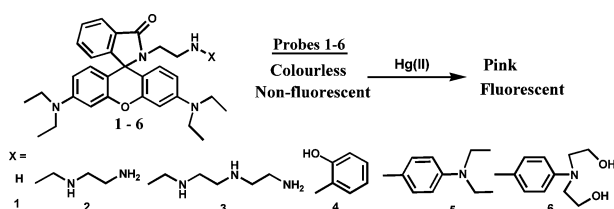


Structure–activity studies of the pelorusides: new congeners and semi-synthetic analogues

A. Jonathan Singh, Mina Razzak, Paul Teesdale-Spittle, Thomas N. Gaitanos, Anja Wilmes, Ian Paterson, Jonathan M. Goodman, John H. Miller and Peter T. Northcote*

New naturally occurring and semi-synthetic pelorusides from the marine sponge *Mycale hentscheli* provide insight into the pharmacophore.

4467

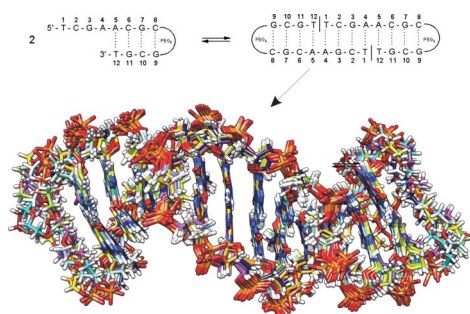


Rhodamine-based probes for metal ion-induced chromo-/fluorogenic dual signaling and their selectivity towards Hg(II) ion

Bamaprasad Bag* and Ajoy Pal

A few substituted amino-derivatives of rhodamine-B (2–6) have exhibited selective and reversible Hg(II)-induced absorption and fluorescence enhancement simultaneously for dual channel signaling.

4481

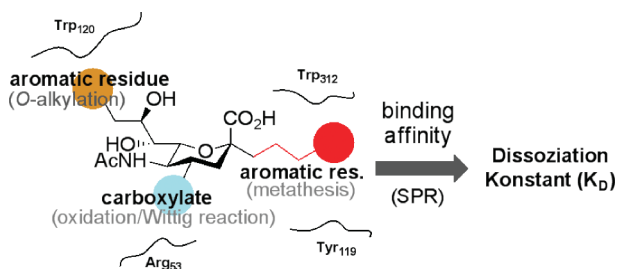


A dumbbell double nicked duplex dodecamer DNA with a PEG₆ tether

Karolina Hyz, Wojciech Bocian, Robert Kawęcki, Elżbieta Bednarek, Jerzy Sitkowski and Lech Kozerski*

A hairpin dodecamer DNA motif with a dangling end composed of four bases was studied in order to find conditions which promote a dumbbell structure as the sole form in solution.

4487



Sialic acid C-glycosides with aromatic residues: Investigating enzyme binding and inhibition of *Trypanosoma cruzi* trans-sialidase

Sebastian Meinke, Andreas Schroven and Joachim Thiem*

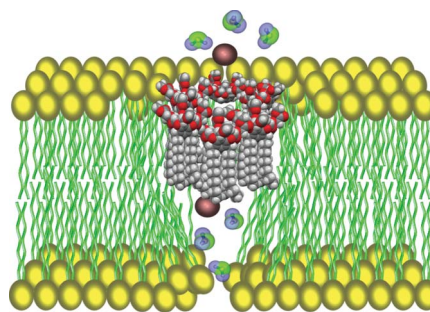
Affinities of α -configured C-sialosides synthesised by cross metathesis were measured against immobilised TcTS by surface plasmon resonance.

4498

Pore formation in phospholipid bilayers by amphiphilic cavitands

Iman Elidrisi, Saeedeh Negin, Pralav V. Bhatt, Thavendran Govender, Hendrick G. Kruger, George W. Gokel* and Glenn E. M. Maguire*

Five new cavitands having *n*-undecyl sidechains are shown to form ion-conducting pores that the evidence suggests result from aggregation within the bilayer.

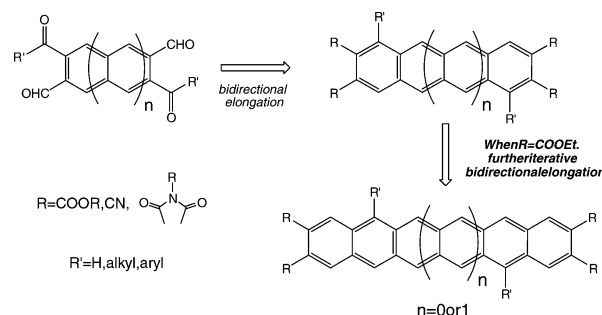


4507

Synthesis of electron deficient acene derivatives via a bidirectional iterative elongation reaction

Yi-Chun Lin, Chih-Hsiu Lin,* Chan-Yu Chen, Shih-Sheng Sun and Bikash Pal

Electron deficient acene derivatives substituted with ester, nitrile, and imide groups are synthesized via a bidirectional elongation protocol.

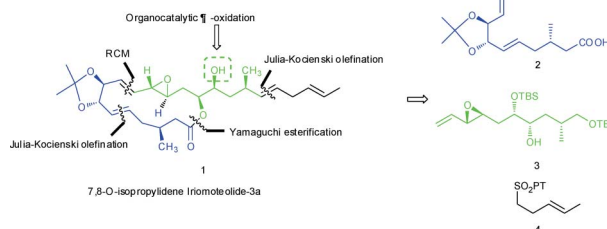


4518

A stereoselective total synthesis of 7,8-*O*-isopropylidene iriomoteolide-3a

Yao Zhang, Lisheng Deng and Gang Zhao*

A concise, and convergent total synthesis of 7,8-*O*-isopropylidene iriomoteolide-3a was achieved in 9 steps and 40% overall yield.

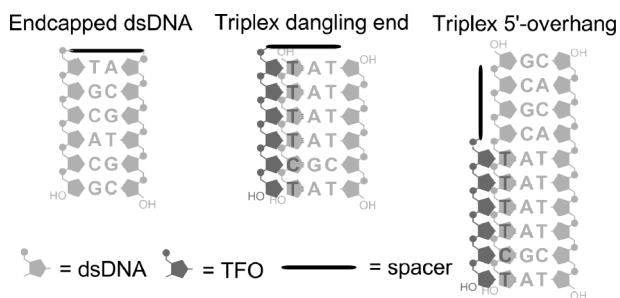


4527

Cationic modified nucleic acids for use in DNA hairpins and parallel triplexes

Niels Bomholt,* Vyacheslav V. Filichev and Erik B. Pedersen

Cationic non-nucleosidic DNA monomers, stabilizing palindromic DNA hairpins and parallel triplexes.



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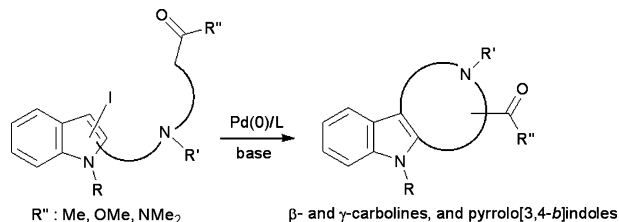
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Intramolecular palladium-catalysed enolate arylation of 2- and 3-iodoindole derivatives for the synthesis of β -carbolines, γ -carbolines, and pyrrolo[3,4-*b*]indoles

Daniel Solé,* M.-Lluïsa Bennasar and Iván Jiménez

The Pd-catalysed intramolecular α -arylation of carbonyl compounds with amino-tethered 2- and 3-iodoindoles provides a useful methodology for the synthesis of indolo-*b*-fused nitrogen heterocycles.

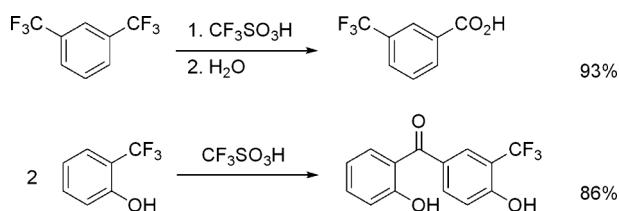


4545

Protolytic defluorination of trifluoromethyl-substituted arenes

Anila Kethe, Adam F. Tracy and Douglas A. Klumpp*

A series of trifluoromethyl-substituted arenes were studied in their reactions with Brønsted superacids.

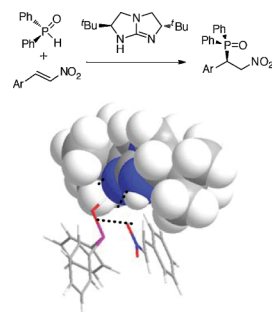


4550

Sequential catalytic role of bifunctional bicyclic guanidine in asymmetric phospho-Michael reaction

Bokun Cho, Choon-Hong Tan and Ming Wah Wong*

The bifunctional mode of activation of bicyclic guanidine catalyst is evidenced in all 3 steps of the catalytic cycle of the guanidine-catalyzed phospho-Michael reaction.

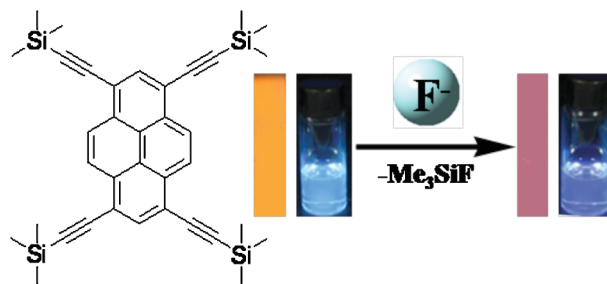


4558

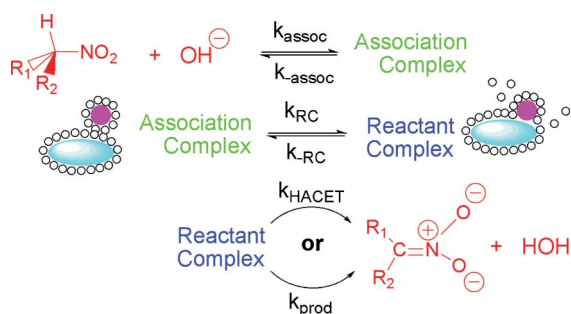
A specific chemodosimeter for fluoride ion based on a pyrene derivative with trimethylsilylethynyl groups

Hua Lu, Qihong Wang, Zhifang Li,* Guoqiao Lai,* Jianxiong Jiang and Zhen Shen*

Pyrene derivative **1** containing four trimethylsilylethynyl groups was investigated as a chromogenic and fluorescence chemodosimeter sensor for fluoride anions.



4563

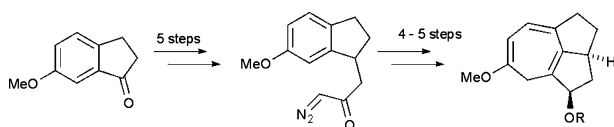


Reactions of the simple nitroalkanes with hydroxide ion in water. Evidence for a complex mechanism

Zhao Li, Jin-Pei Cheng* and Vernon D. Parker*

Intermediates were observed during the complex proton transfer reactions of the simple nitroalkanes, the basis for the “Nitroalkane Anomaly”.

4570

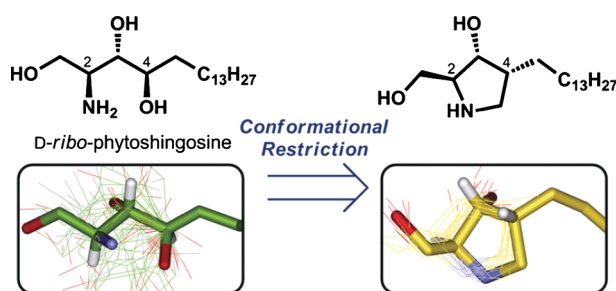


Synthesis of novel molecular probes inspired by harringtonolide

Vinayak Hegde, Marc Campitelli, Ronald J. Quinn and David Camp*

A novel scaffold inspired by the unusual and highly complex natural product, harringtonolide, has been synthesised and enumerated into a small library of derivatives.

4580

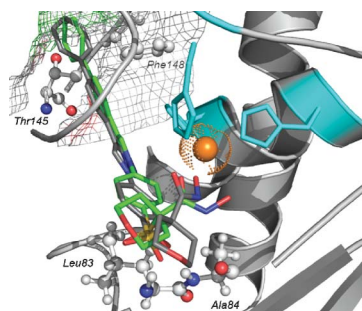


Design and synthesis of pyrrolidine-containing sphingomimetics

Seokwoo Lee, Sukjin Lee, Hyen Joo Park, Sang Kook Lee and Sanghee Kim*

This paper describes conformationally constrained sphingomimetics in which a pyrrolidine moiety is incorporated between the 2-amino group and the C-4 carbon atom of the sphingoid base.

4587



Potent “Clicked” MMP2 Inhibitors: Synthesis, Molecular Modeling and Biological Exploration

Jose María Zapico, Pilar Serra, Josune García-Sanmartín, Kamila Filipiak, Rodrigo J. Carbajo, Anne K. Schott, Antonio Pineda-Lucena, Alfredo Martínez, Sonsoles Martín-Santamaría, Beatriz de Pascual-Teresa* and Ana Ramos*

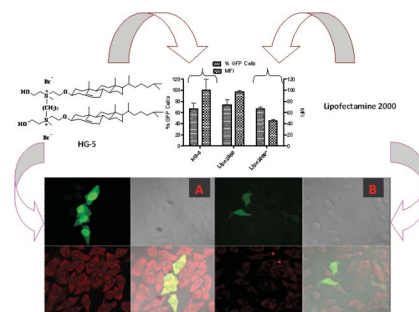
Potent hydroxamate MMP2 inhibitors have been discovered making use of molecular modelling techniques, click chemistry, inhibition assays and NMR experiments.

4600

Syntheses, Transfection Efficacy and Cell Toxicity Properties of Novel Cholesterol-based Gemini Lipids having Hydroxyethyl Head group

Joydeep Biswas, Santosh K. Mishra, Paturu Kondaiah and Santanu Bhattacharya*

HG-5 is a better transfecting agent than Lipofectamine 2000 in the presence of 10% FBS and mean fluorescence intensity shows ~2-fold higher value when HeLa cells were transfected with pEGFP-C3.

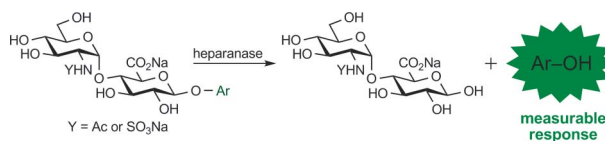


4614

Synthesis of simple heparanase substrates

Andrew G. Pearson, Milton J. Kiefel, Vito Ferro and Mark von Itzstein*

This paper describes the synthesis of glycosyl glucuronides with various aryl aglycones that represent the simplest heparanase substrates reported to date.

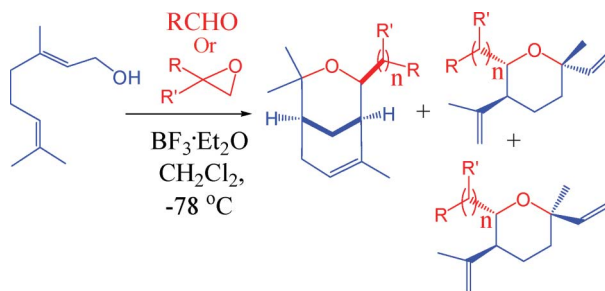


4626

Synthesis of oxabicyclo[3.3.1]nonenes and substituted tetrahydropyrans via (3,5)-oxonium-ene reaction

Pipas Saha, Paramartha Gogoi and Anil K. Saikia*

Oxabicyclo[3.3.1]nonenes and substituted tetrahydropyrans can efficiently be synthesized from geraniol with aldehydes and epoxides via (3,5)-oxonium-ene reaction in moderate yields.

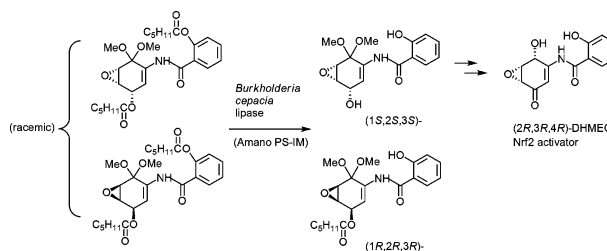


4635

Chemoenzymatic synthesis of (2R,3R,4R)-dehydroxymethylepoxyquinomicin (DHMEQ), a new activator of antioxidant transcription factor Nrf2

Yukihiro Niitsu, Masatoshi Hakamata, Yuko Goto, Toshinori Higashi, Mitsuru Shoji, Takeshi Sugai and Kazuo Umezawa*

(2R,3R,4R)-DHMEQ was efficiently prepared by a new route using lipase, and was shown to activate Nrf2 in a promoter reporter assay.



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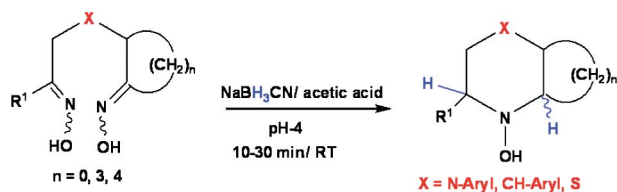
Registered Charity Number 207890

4642

Synthesis of novel *N*-hydroxy heterocycles *via* intramolecular reductive cyclization of diketoximes by NaBH_3CN

Muthupandi Nagaraj, Muthusamy Boominathan, Shanmugam Muthusubramanian* and Nattamai Bhuvanesh

A simple and efficient protocol for the construction of substituted *N*-hydroxy heterocycles through intramolecular reductive cyclization of diketoximes using sodium cyanoborohydride is described

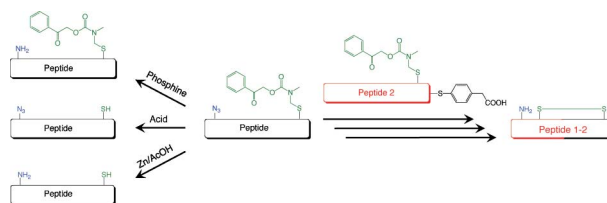


4653

N-Methyl-phenacyloxycarbamidomethyl (Pocam) group: a novel thiol protecting group for solid-phase peptide synthesis and peptide condensation reactions

Hidekazu Katayama,* Yoshiaki Nakahara and Hironobu Hojo*

A novel thiol protecting group (Pocam) was developed and Pocam-containing peptides and peptide thioesters were synthesised.

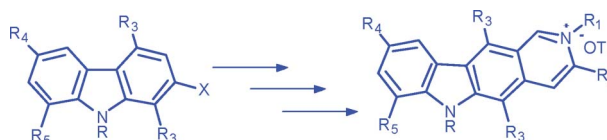


4662

Synthesis of functionalized ellipticinium and ellipticine derivatives *via* electrophilic cyclization

T. Krishna Chaitanya and Rajagopal Nagarajan*

An efficient synthesis of highly functionalized ellipticinium and ellipticine derivatives from the corresponding bromocarbazoles is reported.

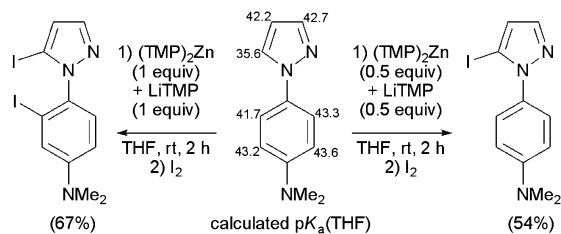


4671

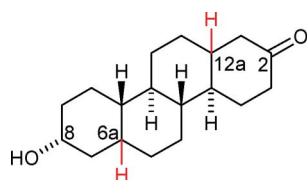
N-aryl pyrazoles: DFT calculations of CH acidity and deprotonative metallation using a combination of lithium and zinc amides

Floris Chevallier,* Yury S. Halauko,* Christelle Pecceu, Ibrahim F. Nassar, To Uyen Dam, Thierry Roisnel, Vadim E. Matulis, Oleg A. Ivashkevich and Florence Mongin*

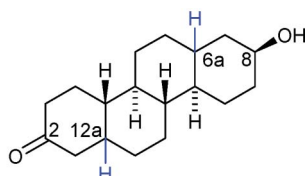
N-(hetero)arylpyrazoles were deprotonated using 1 : 1 LiTMP-(TMP)₂Zn. The reaction outcomes and DFT-calculated CH acidities are discussed.



4685



1: 6a*S*, 12a*R*
 2: 6a*S*, 12a*S*
 3: 6a*R*, 12a*R*
 4: 6a*R*, 12a*S*



ent-1: 6a*R*, 12a*S*
ent-2: 6a*R*, 12a*R*
ent-3: 6a*S*, 12a*S*
ent-4: 6a*S*, 12a*R*

The use of symmetry in enantioselective synthesis: Four pairs of chrysene enantiomers prepared from 19-nortestosterone

Eva Stastna, Nigam P. Rath and Douglas F. Covey*

Four optically pure 2,8-difunctionalized hexadecahydrochrysene diastereomers, and their corresponding optically pure enantiomers were prepared from 19-nortestosterone.

A Healthy, Wealthy, Sustainable World

John Emsley



A Healthy, Wealthy, Sustainable World

John Emsley

Foreword by C David Garner

The world stands at a crossroads. What route to the future should we take? The road to a sustainable future beckons, but what effect will this have on chemistry, which appears so dependent on fossil resources?

This latest book from award-winning science writer, John Emsley, shows how vital chemistry is for producing abundant food, clean water, healing drugs, alternative fuels, and plastics, and how most of the things which a modern society requires now depend on it – and that includes city living and even sport. Can we really produce all that we need from sustainable sources? Given a new generation of bright young chemists he argues that we can.

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