

Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry
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IN THIS ISSUE

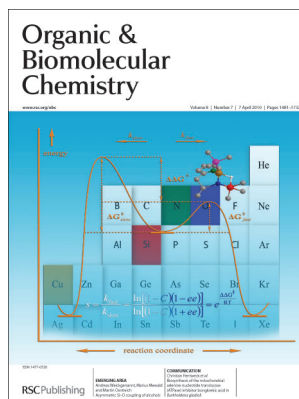
ISSN 1477-0520 CODEN OBCRAK 8(7) 1481–1732 (2010)



Cover

See Werner Hummel *et al.*, pp. 1540–1550.
 Discovery of the bakers' yeast reductase responsible for the reduction of 2,5-hexanedione into enantiopure (2*S*,5*S*)-hexanediol. This diol serves as a building block for the production of various fine chemicals and pharmaceuticals.

Image reproduced by permission of Werner Hummel from *Org. Biomol. Chem.*, 2010, **8**, 1540.



Inside cover

See Martin Oestreich *et al.*, pp. 1497–1504.
 Stereoselective protection of alcohols is achieved through asymmetric Si–O coupling of silicon reagents and either chiral racemic alcohols (kinetic resolution) or prochiral alcohols (desymmetrisation).

Image reproduced by permission of Martin Oestreich from *Org. Biomol. Chem.*, 2010, **8**, 1497.

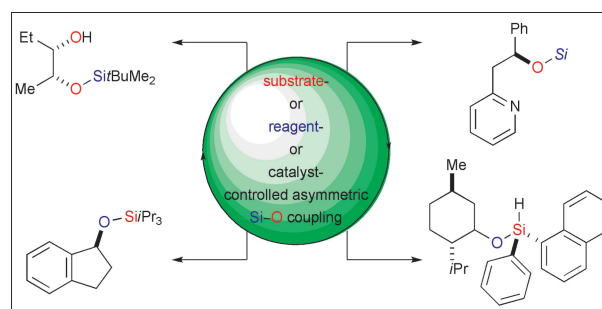
EMERGING AREA

1497

Asymmetric Si–O coupling of alcohols

Andreas Weickgenannt, Marius Mewald and Martin Oestreich*

This Emerging Area summarises about 40 years of investigations directed towards asymmetric Si–O couplings, including substrate-, reagent- and catalyst-controlled approaches.



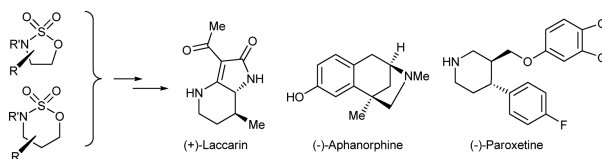
PERSPECTIVE

1505

N-Heterocycle construction *via* cyclic sulfamidates. Applications in synthesis

John F. Bower,* Janjira Rujirawanich and Timothy Gallagher*

1,2- And 1,3-cyclic sulfamidates offer a versatile and effective reactivity profile that is readily harnessed to provide a flexible entry to a wide range of substituted, functionalised and enantiomerically pure *N*-based heterocycles. The scope of this chemistry is illustrated with examples drawn from work within natural products and the pharmaceutical arena.



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Organic & Biomolecular Chemistry

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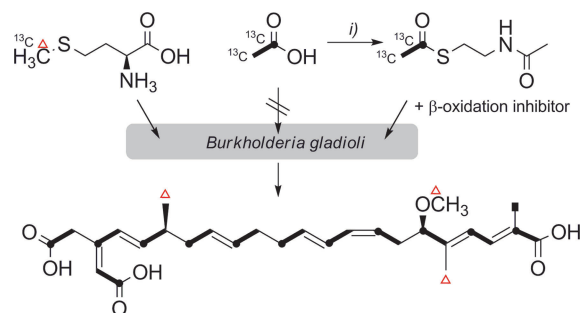
COMMUNICATIONS

1520

Biosynthesis of the mitochondrial adenine nucleotide translocase (ATPase) inhibitor bongkreikic acid in *Burkholderia gladioli*

Barbara Rohm, Kirstin Scherlach and Christian Hertweck*

Biosynthetic studies with ^{13}C -labelled biosynthetic precursors revealed that the infamous, food-related toxin bongkreikic acid is a polyketide with acetate-derived β -branches and a carboxylate terminus derived from the methyl group of acetate.

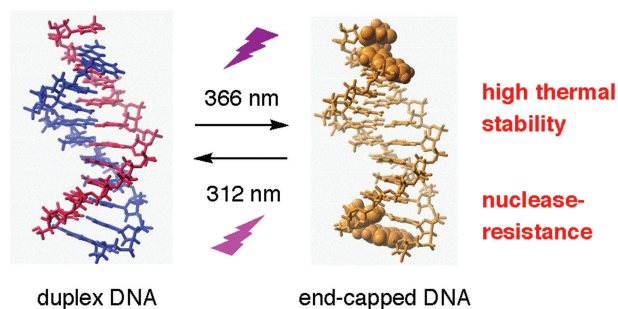


1523

Photoreversible DNA end capping for the formation of hairpin structures

Yoshinaga Yoshimura, Hajime Okada and Kenzo Fujimoto*

We describe a photoreversible DNA end capping *via* 3-cyanovinylcarbazole nucleoside. Doubly end-capped oligodeoxynucleotide (ODN) exhibits increased stability against snake venom phosphodiesterase and shows high thermal stability.

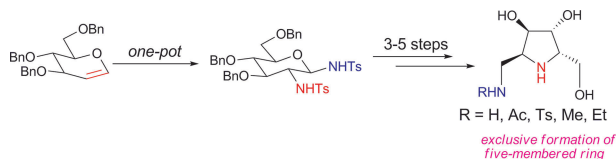


1527

Design and synthesis of new amino-modified iminocyclitols: selective inhibitors of α -galactosidase

Muthupandian Ganesan, Rekhawar V. Madhukarrao and Namakkal G. Ramesh*

A new and short synthesis of hitherto unreported stereo analogue of amino-modified five-membered iminocyclitols, that are selective inhibitors of α -galactosidase, is described.

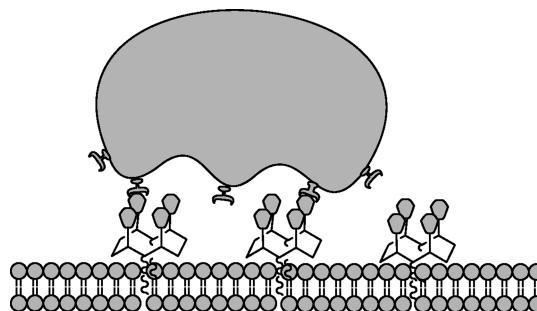


1531

Cell adhesion through clustered ligand on fluid supported lipid bilayers

Ludivine Sandrin, Liliane Coche-Guérente, Amandine Bernstein, Hajra Basit, Pierre Labbé, Pascal Dumy* and Didier Boturyn*

The complementary QCM-D and optical microscopy techniques were used for monitoring cell adhesion on a RGD-functionalized supported lipid bilayer. A critical interligand RGD spacing of nearly 80 nm was estimated for cell adhesion.



Fifth International Symposium on Macrocyclic and Supramolecular Chemistry

ISMSC 2010

June 6 - 10, 2010, Nara (Japan)

Plenary Speakers

Craig Hawker (UC Santa-Barbara, US)
David Leigh (Edinburgh, UK)
Stefan Matile (Geneva, Switzerland)
Alan Rowan (Nijmegen, Netherlands)
Mitsuhiko Shionoya (Tokyo, Japan)

Invited Speakers

Takuzo Aida / Cornelia Bohne / David Dearden /
Harry Anderson / Lee Cronin / Philip Gale /
Shinji Inagaki / Kimoon Kim / Nobuo Kimizuka /
Hiroshi Kitagawa / Jeffery Moore / Achim Müller /
Colin Nuckolls / Hiroyuki Noji / V. Ramamurthy /
Julius Rebek, Jr. / Jean-Pierre Sauvage / Jonathan Sessler /
George Shimizu / Fraser Stoddart / James Tucker /
Thomas R. Ward / Omar Yaghi / Kimihisa Yamamoto /
Vivian Yam / Nobuhiko Yui

Important Deadlines

Early Registration: April, 15th
Regular Registration: May, 15th
Abstract submission: May, 15th

In 2006, the International Symposium on Macrocyclic Chemistry (established in 1977) and the International Symposium on Supramolecular Chemistry (established in 1980) were merged in a constructive way to establish ISMSC. Since then, ISMSC has been the largest symposium in the fields of macrocyclic chemistry and supramolecular chemistry.



For registration, abstract submission
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Nara is the ancient capital of Japan. The year 2010 is the 1300th anniversary of Nara Capital. So, various events will be held in Nara to commemorate the 1300th anniversary.

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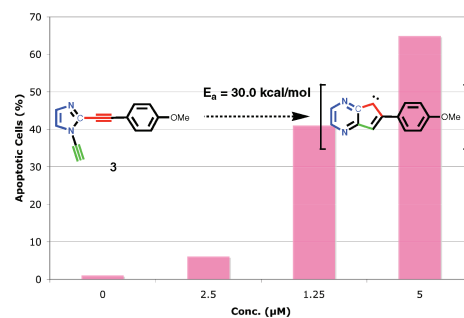
COMMUNICATIONS

1535

Cyclization kinetics and biological evaluation of an anticancer 1,2-dialkynylimidazole

Christophe Laroche, Jing Li, Cristina Gonzales, Wendi M. David and Sean M. Kerwin*

An improved procedure for the synthesis of 1-alkynylimidazole derivatives has been employed to prepare sufficient quantities of **3** for biological evaluation. The 1,2-dialkynylimidazole **3** is cytotoxic against a wide range of cancer cells and induces apoptosis in A549 cells.



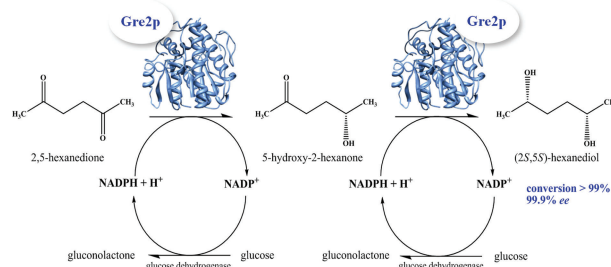
PAPERS

1540

Highly efficient and stereoselective biosynthesis of (2*S*,5*S*)-hexanediol with a dehydrogenase from *Saccharomyces cerevisiae*

Marion Müller, Michael Katzberg, Martin Bertau and Werner Hummel*

The dehydrogenase which is responsible for the stereoselective reduction of 2,5-hexanedione in bakers' yeast was identified. Enzymatic synthesis now enables a highly efficient synthesis route to (2*S*,5*S*)-hexanediol. The high space-time yield make the process transferable to an industrial scale.

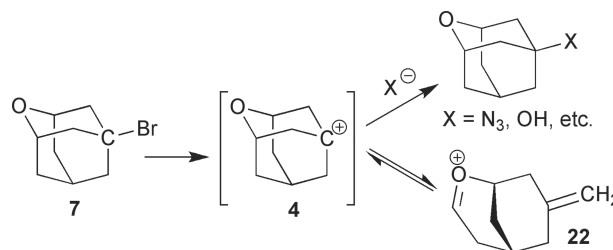


1551

Searching for intermediates in Prins cyclisations: the 2-oxa-5-adamantyl carbocation

Roger W. Alder,* Fabrizio Carta, Christopher A. Reed, Irina Stoyanova and Christine L. Willis

2-Oxa-5-adamantyl carbocation **4** is a viable intermediate in several S_N1 reactions of 5-bromo-2-oxaadamantane but attempts to observe **4** by NMR methods failed, probably because **4** undergoes reversible ring opening to **22**, which is destroyed under superacid conditions.

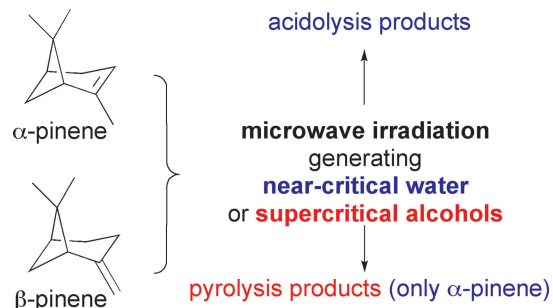


1560

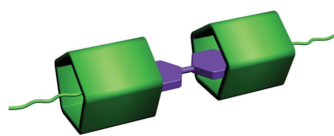
Fate of monoterpenes in near-critical water and supercritical alcohols assisted by microwave irradiation

Tony Szuppa, Achim Stolle* and Bernd Ondruschka

The behaviour of α - and β -pinene in near-critical water and supercritical alcohols generated under closed-vessel conditions using a microwave was investigated, revealing significant differences in product distribution and reactivity.



1568



2:1 External Complex with Alkyl Substituted Paraquats



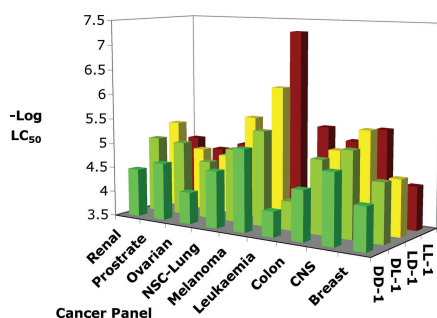
1:1 Pseudorotaxane Complex with 1,4-Bis(pyridinium)butane

Complex interactions of pillar[5]arene with paraquats and bis(pyridinium) derivatives

Chunju Li,* Qianqian Xu, Jian Li, Feina Yao and Xueshun Jia*

The pillar[5]arene host forms 2 : 1 external complexes with alkyl-substituted paraquats, and it forms 1 : 1 pseudorotaxane-type inclusion complexes with methylene $[-(\text{CH}_2)_n-]$ connected bis(pyridinium) derivatives.

1577

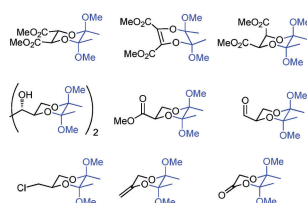


Triple molecular target approach to selective melanoma cytotoxicity

Edward B. Skibo,* Akmal Jamil, Brittany Austin, Douglas Hansen and Armand Ghodousi

Phenylalanine-linked pyrrolo[1,2-*a*]benzimidazole **LL1** was successfully designed to target melanoma cells *in vitro*; the design utilised three molecular targets: a phenylalanine pump, the reducing enzyme DT-diaphorase, and IMP dehydrogenase.

1588



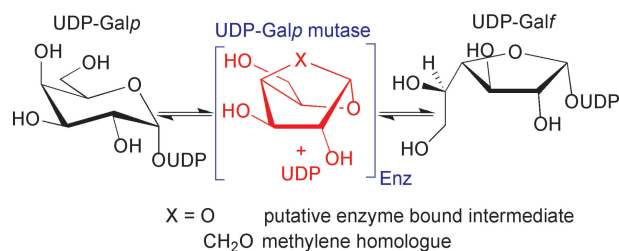
- continuous processing
- superior yields
- in-line purification

The continuous flow synthesis of butane-2,3-diacetal protected building blocks using microreactors

Catherine F. Carter, Ian R. Baxendale, John B. J. Pavey and Steven V. Ley*

The continuous flow syntheses of a family of butane-2,3-diacetal protected building blocks has been achieved using microreactors in concert with solid supported reagents and scavengers.

1596

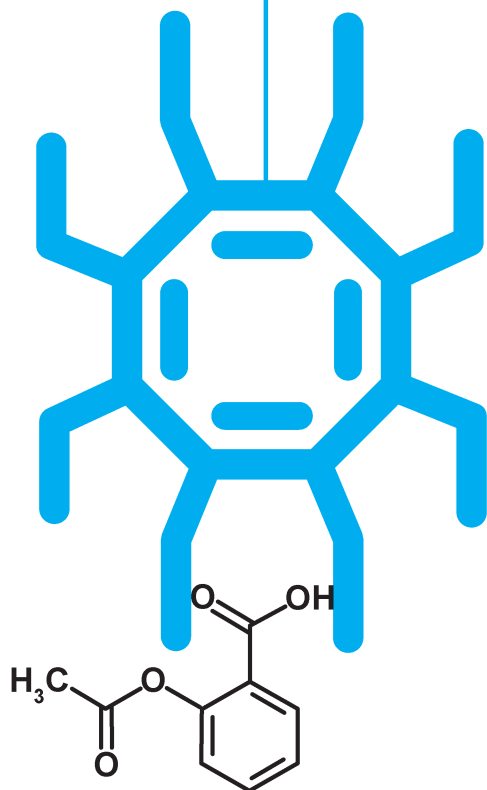


The UDP-Galp mutase catalyzed isomerization: synthesis and evaluation of 1,4-anhydro-β-D-galactopyranose and its [2.2.2] methylene homologue

Ali Sadeghi-Khomami, Tatiana J. Forcada, Claire Wilson, David A. R. Sanders and Neil R. Thomas*

The synthesis of 1,4-anhydro-β-D-galactopyranose (1,5-anhydro-α-D-galactofuranose), a proposed intermediate in the ring contraction isomerisation catalyzed by UDP-galactopyranose mutase, together with its [2.2.2] bicyclic methylene homologue, synthesised as a possible competitive inhibitor or alternative substrate, are reported.

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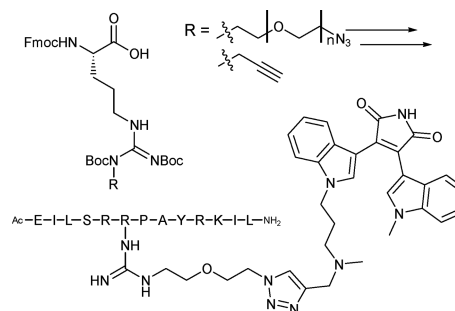
PAPERS

1629

Preparation of novel alkylated arginine derivatives suitable for click-cycloaddition chemistry and their incorporation into pseudosubstrate- and bisubstrate-based kinase inhibitors

Jeroen van Ameijde, Alex J. Poot, Loek T. M. van Wandelen, Angelique E. M. Wammes, Rob Ruijtenbeek, Dirk T. S. Rijkers and Rob M. J. Liskamp*

Novel modified arginine residues suitable for 'click' chemistry and their use in a PKC inhibitor are described.

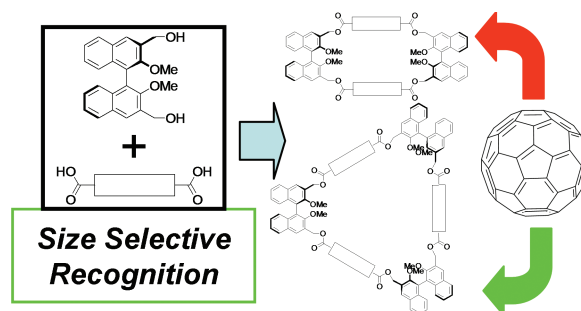


1640

Structurally-variable, rigid and optically-active D_2 and D_3 macrocycles possessing recognition properties towards C_{60}

Carmine Coluccini, Daniele Dondi, Marco Caricato, Angelo Taglietti, Massimo Boiocchi and Dario Pasini*

A straightforward route to chiral macrocycles is described. The larger [3 + 3] macrocycles possess the right cavity size for the complexation of C_{60} , with switchable stoichiometries in relation to the spacer shapes defining the cavities of the cyclic structures.

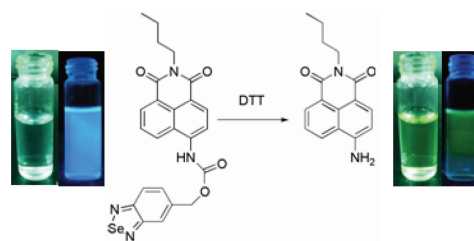


1650

A highly selective ratiometric fluorescent probe for 1,4-dithiothreitol (DTT) detection

Baocun Zhu, Xiaoling Zhang,* Hongying Jia, Yamin Li, Haipeng Liu and Weihong Tan*

A highly selective ratiometric fluorescent probe for 1,4-dithiothreitol (DTT) was designed and synthesized, which displays a 66 nm red-shift of fluorescence emission and the color changes from colorless to jade-green upon reaction with DTT.

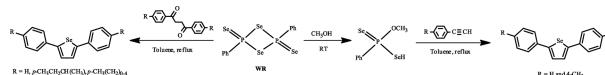


1655

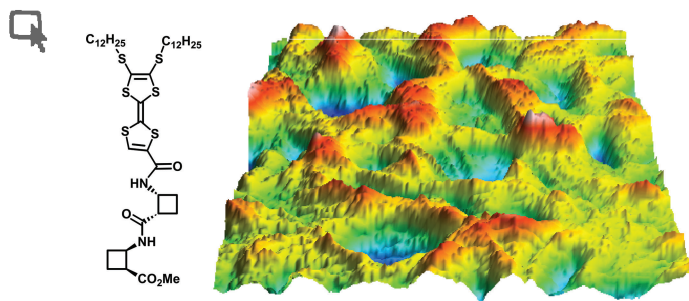
Synthesis of novel 2,5-diarylselenophenes from selenation of 1,4-diarylbutane-1,4-diones or methanol/arylacetylenes

Guoxiong Hua, John B. Henry, Yang Li, Andrew R. Mount, Alexandra M. Z. Slawin and J. Derek Woollins*

2,5-Diarylselenophenes can be prepared by reaction of *O*-methyl *Se*-hydrogen phenylphosphonodiselenoate with arylacetylenes or by direct reaction of Woollins' reagent with 1,4-diarylbutane-1,4-diones.



1661

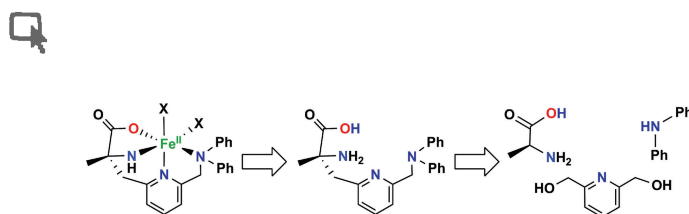


Use of unnatural β -peptides as a self-assembling component in functional organic fibres

Elisabeth Torres, Josep Puigmartí-Luis, Ángel Pérez del Pino, Rosa M. Ortuño* and David B. Amabilino*

Supramolecular fibres are formed by a homochiral synthetic dipeptide incorporating two cyclobutyl rings. Current-sensing AFM shows that once doped, films of the material are capable of conducting electricity.

1666

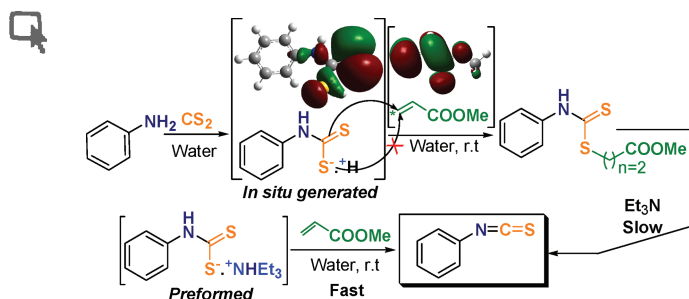


Design and synthesis of a tetradentate '3-amine-1-carboxylate' ligand to mimic the metal binding environment at the non-heme iron(II) oxidase active site

Victoria J. Dungan, Yannick Ortin, Helge Mueller-Bunz and Peter J. Rutledge*

Non-heme iron(II) oxidases (NHIOs) promote a raft of interesting oxidation reactions *in vivo*. Can we replicate the iron binding environment of the NHIO active site to create biomimetic small-molecule systems that promote hydrocarbon oxidation *in vitro*?

1674

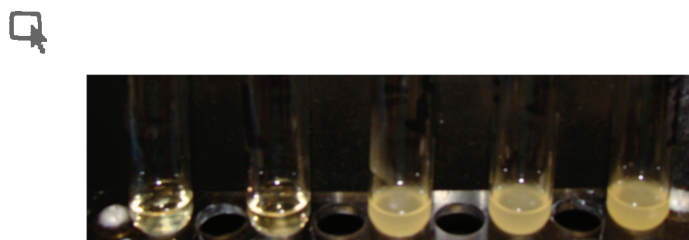


The thiocarbonyl 'S' is softer than thiolate 'S': A catalyst-free one-pot synthesis of isothiocyanates in water

Latonglila Jamir, Abdur Rezzak Ali, Harisadhan Ghosh, Francis A. S. Chipem and Bhisma K. Patel*

Treatment of the preformed or the *in situ* generated aryl/alkyl dithiocarbamates triethylammonium salt with methyl acrylate in an aqueous medium gave solely arylisothiocyanate, whereas the *in situ* generated aryl dithiocarbamic acid yielded exclusively the *thia*-Michael adduct.

1679



The effects of tryptophan and hydrophobicity on the structure and bioactivity of novel indolicidin derivatives with promising pharmaceutical potential

Aaron P. Podorieszsch and Heidi E. K. Huttunen-Hennelly*

We have created novel indolicidin derivatives that exhibit promising pharmaceutical potential, strong antimicrobial and low hemolytic activity. Furthermore, we report the first activity observed against *Candida albicans*, a common pathogen causing yeast infections and oral thrush.

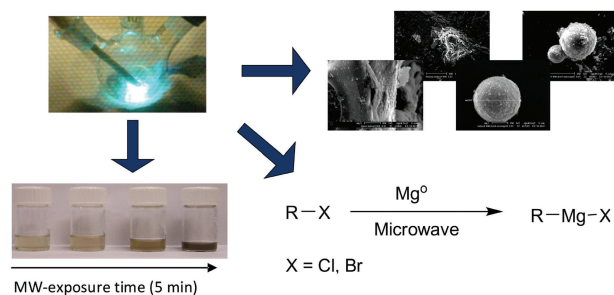
PAPERS

1688

Microwave-induced electrostatic etching: generation of highly reactive magnesium for application in Grignard reagent formation

Bastiaan H. P. van de Kruijs, Mark H. C. L. Dressen, Jan Meuldijk, Jef A. J. M. Vekemans and Lumbertus A. Hulshof*

Microwave-induced electrical discharges influence the surface and, therefore, the reactivity in Grignard reagent formation of magnesium turnings.

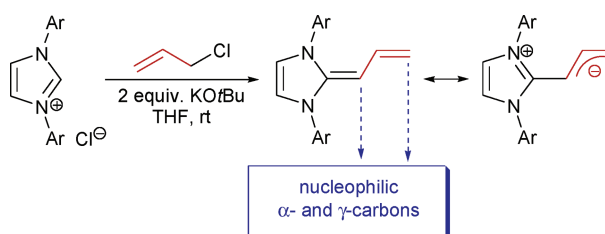


1695

On new N-heterocyclic carbene derived alkylidene imidazolines

Christiane E. I. Knappke, Jörg M. Neudörfel and Axel Jacobi von Wangelin*

Oxygen-free: Substitution of allyl halides with N-heterocyclic carbenes results in the formation of nucleophilic 1,1-diamino-1,3-dienes which are deoxy-analogues of the conceptually related Breslow-type homoenolates and exhibit significant zwitterionic character.

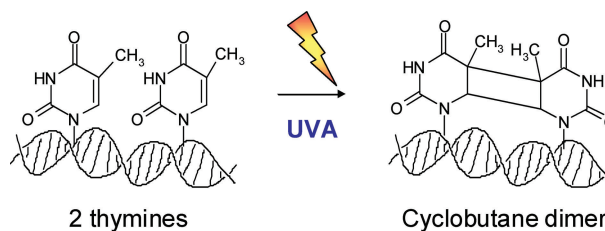


1706

UVA-induced cyclobutane pyrimidine dimers in DNA: a direct photochemical mechanism?

Stéphane Mouret, Coralie Philippe, Jocelyne Gracia-Chantegrel, Akos Banyasz, Szilvia Karpati, Dimitra Markovitsi and Thierry Douki*

Cyclobutane pyrimidine dimers produced in DNA upon UVA irradiation arise from a direct photochemical mechanism rather than a photosensitized process.

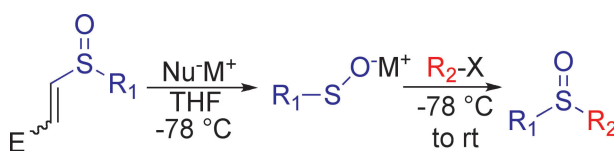


1712

Nucleophilic attack of 2-sulfinyl acrylates: A mild and general approach to sulfenic acid anions

Suneel P. Singh, Jennifer S. O'Donnell and Adrian L. Schwan*

A stereospecific addition/elimination of 2-sulfinyl acrylates using various nucleophiles is demonstrated as a general protocol for alkane- and arenesulfenate generation. A variety of sulfoxides are prepared through alkylation chemistry.



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

13 - 15 September 2010
Durham University, UK
www.rsc.org/DD12



Call for posters now open

Organised jointly by the Dalton Division and Organic Division, DD12 will bring together the organic, organometallic and inorganic (coordination chemistry) communities from academia and industry to discuss the current state of the art, the development and future of late metal-catalysed cross-coupling strategies involving C-X and/or C-H bonds.

The meeting will highlight the importance of catalytic bond activation in cross-coupling chemistry. The latest research will be presented and discussed.

Topics

- Synthetic chemistry (including applications)
- Inorganic and organometallic chemistry
- Reaction mechanism (physical organic and organometallic chemistry)
- Transition metal catalysis
- Applications of C-H and C-X bond activation in organic synthesis

Keynote speakers

Jennifer Love
*The University of British Columbia,
Canada*

William D. Jones
University of Rochester, USA

Aiwen Lei
Wuhan University, China

Zhang-jie Shi
Peking 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*

Offers of contributed papers related to the listed themes for poster presentation are invited by 16 July 2010.

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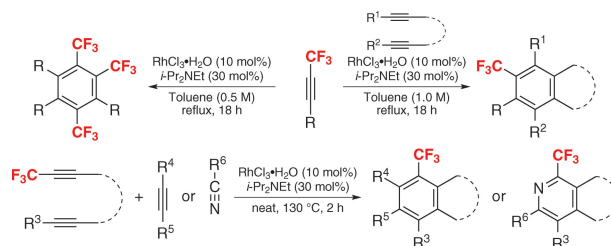
Registration will open in spring 2010.

1718

Rhodium-catalyzed [2+2+2] cycloaddition of various fluorine-containing alkynes—novel synthesis of multi-substituted fluoroalkylated aromatic compounds

Tsutomu Konno,* Kazuki Moriyasu, Ryoko Kinugawa and Takashi Ishihara

Various fluorinated internal alkynes underwent a smooth rhodium-catalyzed [2+2+2] cycloaddition to afford multi-substituted fluoroalkylated aromatic compounds in good to high yields.

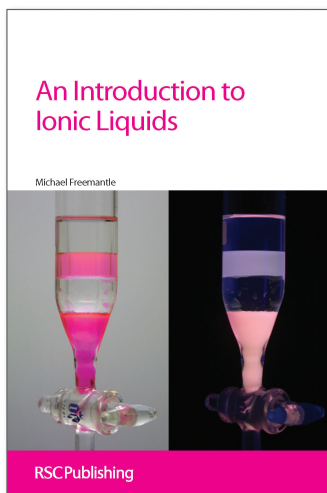
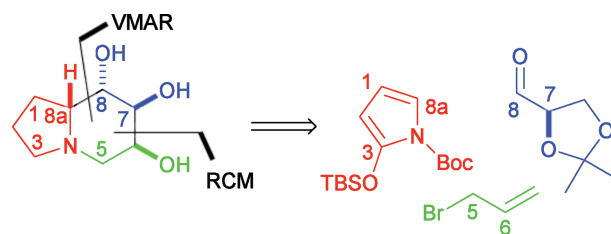


1725

Asymmetric total synthesis of 1-deoxy-7,8-di-*epi*-castanospermine

Vincenzo Zambrano,* Gloria Rassu, Annamaria Roggio, Luigi Pinna, Franca Zanardi, Claudio Curti, Giovanni Casiraghi* and Lucia Battistini*

An efficient, stereocontrolled synthesis of 1-deoxy-7,8-di-*epi*-castanospermine has been developed involving a vinylogous Mukaiyama aldol reaction (VMAR) and an ene-ene ring closing metathesis reaction (RCM) as key steps.



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Michael Freemantle

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