

Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

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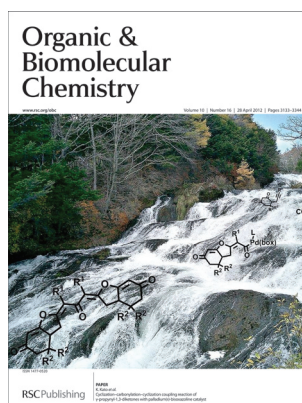
ISSN 1477-0520 CODEN OBCRAK 10(16) 3133–3344 (2012)



Cover

See B. Zajc *et al.*,
pp. 3164–3167.

A facile building block approach allows synthesis of 1-substituted-1-fluoroethenes under mild conditions. The authors thank Satish Lakshman (Pixiedust Design) for the cover art design. Image reproduced by permission of Barbara Zajc from *Organic & Biomolecular Chemistry*, 2012, **10**, 3164.



Inside cover

See K. Kato *et al.*,
pp. 3192–3194.

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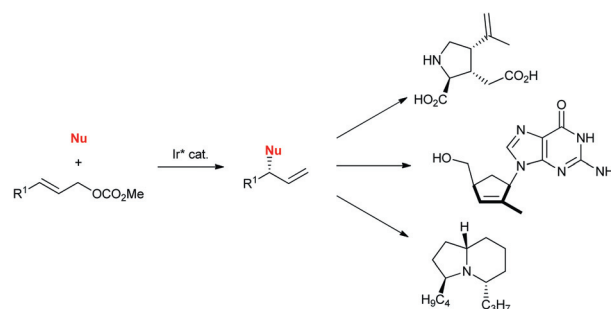
PERSPECTIVE

3147

Recent advances and applications of iridium-catalysed asymmetric allylic substitution

Paolo Tosatti,* Adam Nelson and Stephen P. Marsden

The development of chiral iridium catalysts for asymmetric allylic substitution reactions and their application to the synthesis of natural products and other biologically relevant compounds is reviewed.



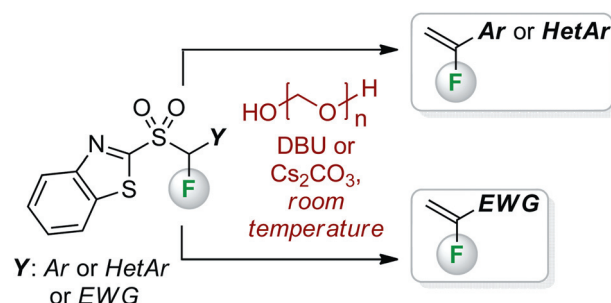
COMMUNICATIONS

3164

Expedient synthesis of α -substituted fluoroethenes

Samir K. Mandal, Arun K. Ghosh, Rakesh Kumar and Barbara Zajc*

Reactions of paraformaldehyde with fluorinated Julia–Kocienski reagents yield 1-substituted 1-fluoroethenes under mild conditions.



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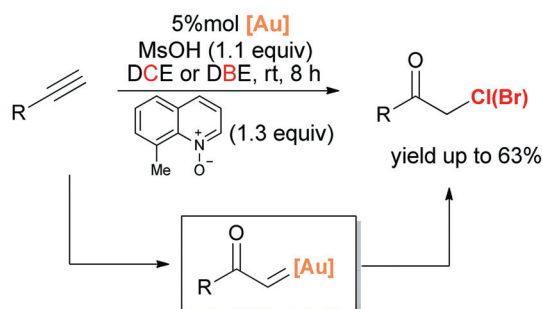
COMMUNICATIONS

3168

Electrophilicity of α -oxo gold carbene intermediates: halogen abstractions from halogenated solvents leading to the formation of chloro/bromomethyl ketones

Weimin He, Longyong Xie, Yingying Xu, Jiannan Xiang* and Liming Zhang*

α -Oxo gold carbenes generated *via* intermolecular oxidation of terminal alkynes can effectively abstract halogen from halogenated solvents.

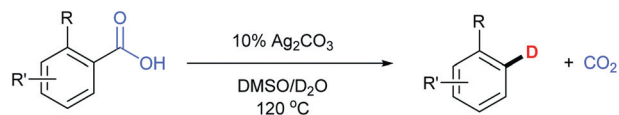


3172

Selective deuteration of (hetero)aromatic compounds *via* deuterio-decarboxylation of carboxylic acids

Rachel Grainger, Arif Nikmal, Josep Cornella and Igor Larrosa*

A practical, mild and highly selective protocol for the monodeuteration of a variety of arenes and heteroarenes is presented. Catalytic amounts of Ag(I) salts in DMSO/D₂O are shown to facilitate the deuterio-decarboxylation of *ortho*-substituted benzoic and heteroaromatic α -carboxylic acids in high yields with excellent levels of deuterium incorporation.

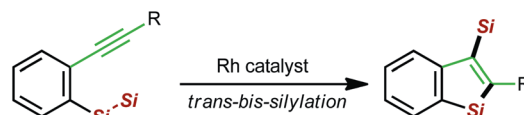


3175

Rhodium-catalysed intramolecular *trans*-bis-silylation of alkynes to synthesise 3-silyl-1-benzosiloles

Takanori Matsuda* and Yoichiro Ichioka

Intramolecular addition of a Si–Si bond across a C–C triple bond occurs in a *trans* fashion in the presence of rhodium(I) catalysts. The *trans*-bis-silylation reaction of (2-alkynylphenyl)disilanes affords 3-silyl-1-benzosiloles.

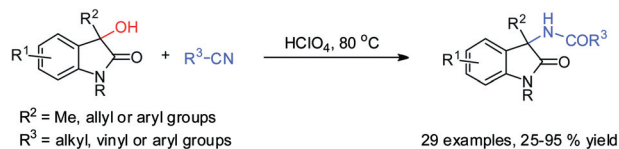


3178

A catalytic metal-free Ritter reaction to 3-substituted 3-aminoxindoles

Feng Zhou, Miao Ding and Jian Zhou*

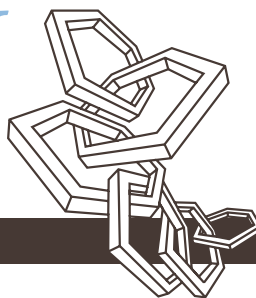
The first Ritter reaction of 3-substituted-3-hydroxyoxindoles with nitriles, catalyzed by HClO₄, is developed, enabling the synthesis of 3-aminoxindoles in rich diversity.



BOSS XIII

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 - Prof. Paul A. WENDER (Stanford University, USA)
 - Prof. Ming Hua XU (Shanghai Institute of Materia Medica, China)
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- **Poster sessions**
- **Exhibition**
- **Social activities**

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Janssen Pharmaceutica Prize for Creativity in Organic Synthesis

Prof. Ilan Marek (Technion - Israel Institute of Technology, Israel) has been designated by the Jury as the winner of the 2012 Janssen Pharmaceutica Prize for Creativity in Organic Synthesis, established in order to honour Dr Paul Janssen, founder of Janssen Pharmaceutica. The award will be delivered after the lecture of Prof. Marek on Thursday July 19, 2012.

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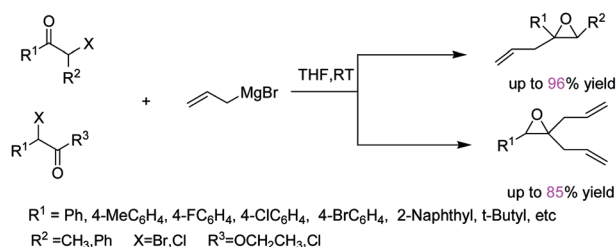
COMMUNICATIONS

3182

An efficient synthetic method for allyl-epoxides *via* allylation of α -haloketones or esters with allylmagnesium bromide

Liyuan Fan, Min Zhang and Songlin Zhang*

The manuscript describes the synthesis of allyl-epoxides and diallyl-epoxides by allylation of α -haloketones and esters with allylmagnesium bromide in mild conditions.

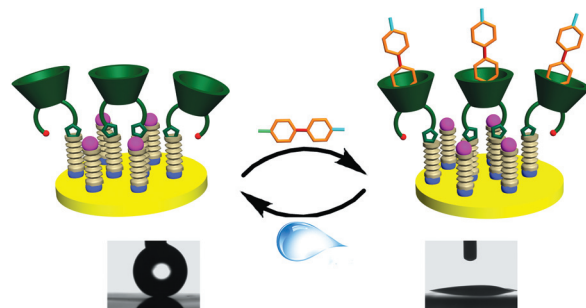


3185

Design of switchable wettability sensor for paraquat based on clicking calix[4]arene

Guifen Zhang, Xiaolei Zhu, Fajun Miao, Demei Tian and Haibing Li*

A calix[4]arene acetylene-modified gold surface is constructed *in situ* via click chemistry and used for selective recognition of paraquat by a wettability switch. Impedance measurements show that it can also recognise paraquat with high sensitivity. The host-guest inclusion-based recognition is studied computationally and a possible mechanism analyzed.

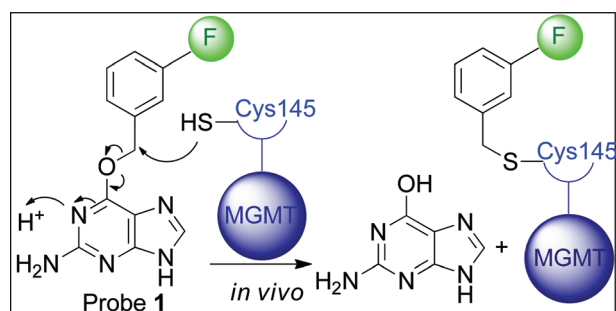


3189

A mechanism-based fluorescent probe for labeling O^6 -methylguanine-DNA methyltransferase in live cells

Xin Li, Shijing Qian, Lin Zheng, Bo Yang, Qiaojun He and Yongzhou Hu*

A mechanism-based fluorescent probe for detecting active O^6 -methylguanine-DNA methyltransferase in live cells has been developed.



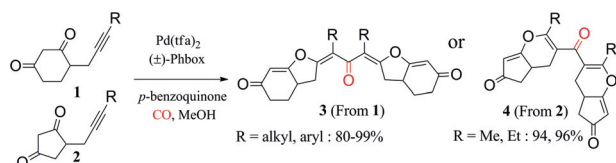
PAPERS

3192

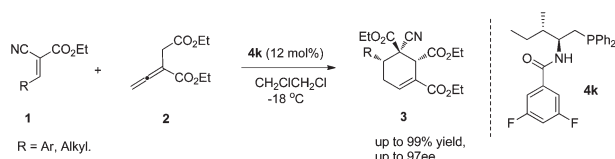
Cyclization–carbonylation–cyclization coupling reaction of γ -propynyl-1,3-diketones with palladium(II)-bisoxazoline catalyst

Taichi Kusakabe, Yasuko Kawai, Rong Shen, Tomoyuki Mochida and Keisuke Kato*

Cyclization–carbonylation–cyclization coupling reactions (CCC-coupling reaction) of γ -propynyl-1,3-diketones catalyzed by (box)Pd^{II} complexes afforded symmetrical ketones possessing two oxabicyclic groups in moderate to excellent yields.



3195

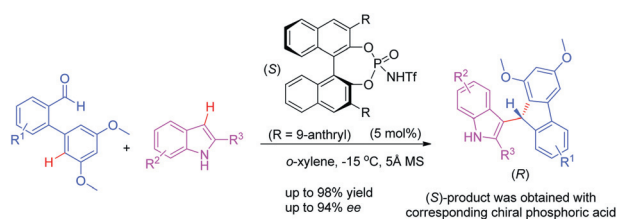


Highly enantioselective [4 + 2] cycloadditions of allenoates and dual activated olefins catalyzed by *N*-acyl aminophosphines

Hua Xiao, Zhuo Chai, Dongdong Cao, Hongyu Wang, Jinghao Chen and Gang Zhao*

An asymmetric organocatalytic [4 + 2] cycloaddition between α -substituted allenoates and 2-cyano acrylates using bifunctional *N*-acyl aminophosphine catalysts is described.

3202

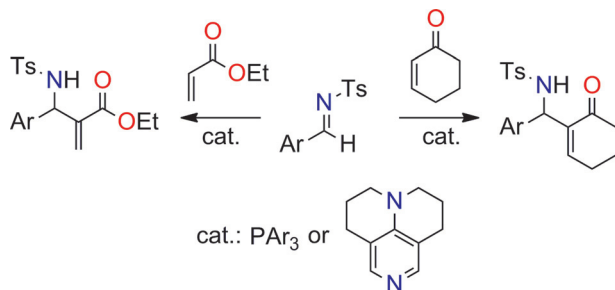


Enantioselective synthesis of fluorene derivatives by chiral *N*-triflyl phosphoramidate catalyzed double Friedel–Crafts alkylation reaction

Shou-Guo Wang, Long Han, Mi Zeng, Feng-Lai Sun, Wei Zhang and Shu-Li You*

Tandem double Friedel–Crafts reaction between indoles and 2-formylbiphenyls by chiral *N*-triflyl phosphoramidate was realized, affording (3-indolyl) fluorenes with excellent enantioselectivity.

3210

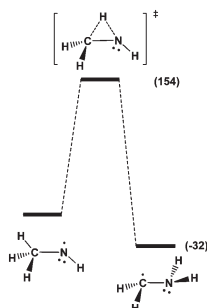


The aza-Morita–Baylis–Hillman reaction of electronically and sterically deactivated substrates

Christoph Lindner, Raman Tandon, Yinghao Liu, Boris Maryasin and Hendrik Zipse*

Kinetic measurements and theoretical studies have been combined to develop highly active catalysts for the aza-Morita–Baylis–Hillman reaction of electronically or sterically deactivated substrates.

3219



Hydrogen tunnelling influences the isomerisation of some small radicals of interstellar importance. A theoretical investigation

Tianfang Wang and John H. Bowie*

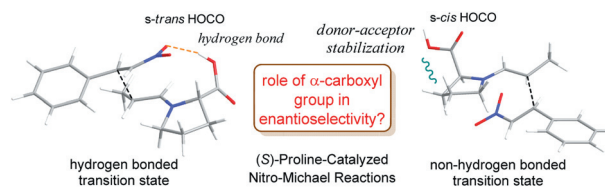
Hydrogen atom isomerisations within five radical systems (*i.e.*, $CH_3\cdot NH\cdot / CH_2NH\cdot$; $CH_3O\cdot / CH_2OH\cdot$; $\cdot CH_2SH / CH_3S\cdot$; $CH_3CO_2\cdot / CH_2CO_2H\cdot$; and $HOCH_2CH_2O\cdot / HO\cdot CHCH_2OH$) have been studied *via* quantum-mechanical hydrogen tunnelling through reaction barriers; *e.g.* $CH_3\cdot NH\cdot \rightarrow \cdot CH_2NH_2$.

3229

(S)-Proline-catalyzed nitro-Michael reactions: towards a better understanding of the catalytic mechanism and enantioselectivity

Hui Yang and Ming Wah Wong*

A non-hydrogen bonded type of transition state is essential to understand the observed enantioselectivities of the proline-catalyzed nitro-Michael reactions of aldehyde and ketone.

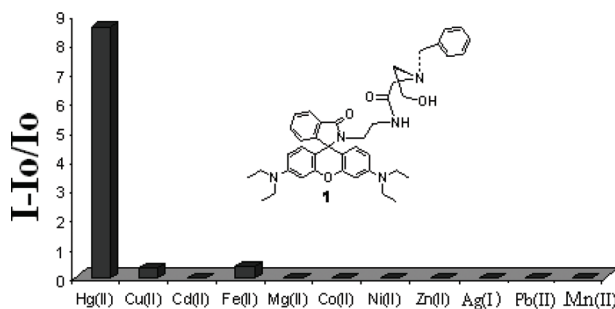


3236

A rhodamine appended tripodal receptor as a ratiometric probe for Hg²⁺ ions

Kumares Ghosh,* Tanmay Sarkar and Asmita Samadder

A new rhodamine appended tripodal receptor **1** has been designed and synthesized.

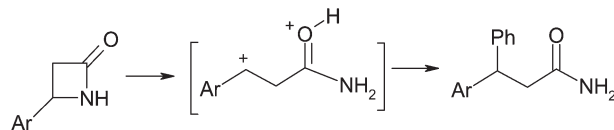


3244

The acid-mediated ring opening reactions of α -aryl-lactams

Frank D. King* and Stephen Caddick

α -Arylazetid-2-ones undergo TfOH- or AlCl₃-mediated ring opening in benzene to give 3-aryl-3-phenyl-propionamides *via* the dication. Neutralisation of the dication gives cinnamamides. Further reaction with benzene gives 3,3-diphenyl-propionamide.

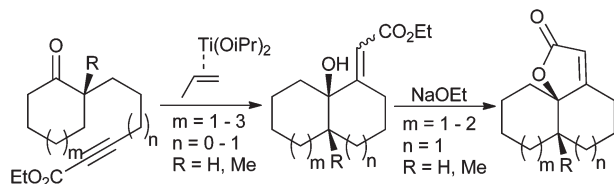


3253

Intramolecular reductive ketone-alkynoate coupling reaction promoted by (η^2 -propene)titanium

Christian Schäfer, Michel Miesch* and Laurence Miesch*

Intramolecular reductive coupling of cycloalkanones tethered to alkynoates in the presence of (η^2 -propene)titanium diastereoselectively provided hydroxy-esters which led to angularly fused unsaturated tricyclic lactones.



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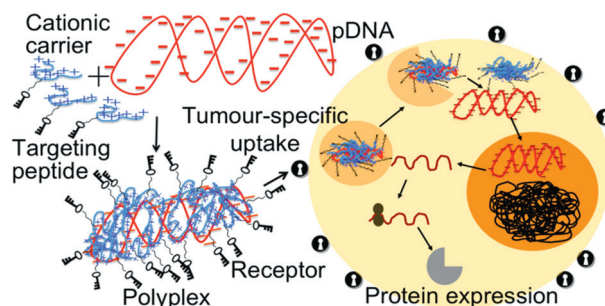
PAPERS

3258

Solid-phase-assisted synthesis of targeting peptide–PEG–oligo(ethane amino)amides for receptor-mediated gene delivery

Irene Martin, Christian Dohmen, Carlos Mas-Moruno, Christina Troiber, Petra Kos, David Schaffert, Ulrich Lächelt, Meritxell Teixidó, Michael Günther, Horst Kessler, Ernest Giralt* and Ernst Wagner*

Oligoaminoamides containing polyethylene glycol and integrin or transferrin receptor ligands are precise carriers for DNA transfer into tumor cells.

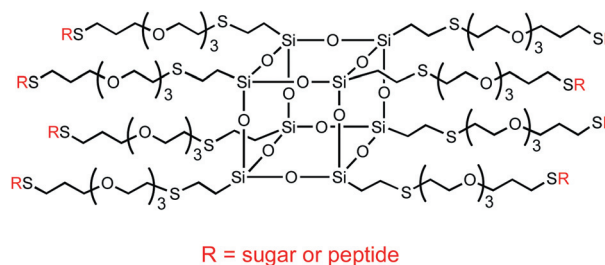


3269

Glycoside and peptide clustering around the octasilsesquioxane scaffold via photoinduced free-radical thiol–ene coupling. The observation of a striking glycoside cluster effect

Mauro Lo Conte, Samuele Staderini, Angela Chambery, Nathalie Berthet, Pascal Dumy, Olivier Renaudet, Alberto Marra* and Alessandro Dondoni*

Structure of multivalent conjugates prepared by thiol–ene coupling of sugar and peptide thiols with a PEGylated octasilsesquioxane functionalized with terminal allyl groups.

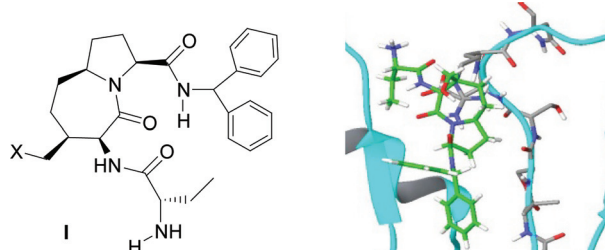


3278

A NMR and computational study of Smac mimics targeting both the BIR2 and BIR3 domains in XIAP protein

Donatella Potenza,* Laura Belvisi,* Francesca Vasile, Elisabetta Moroni, Federica Cossu and Pierfausto Seneci

In this paper we report an extensive computational and NMR analysis of small ligands (Smac mimics) complexed with different constructs of XIAP.

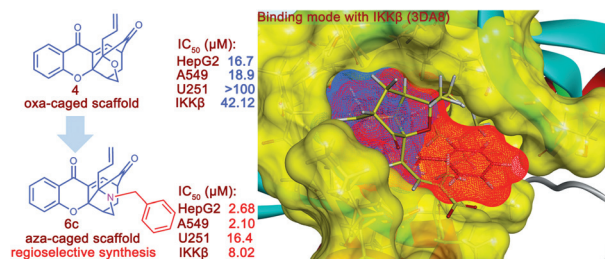


3288

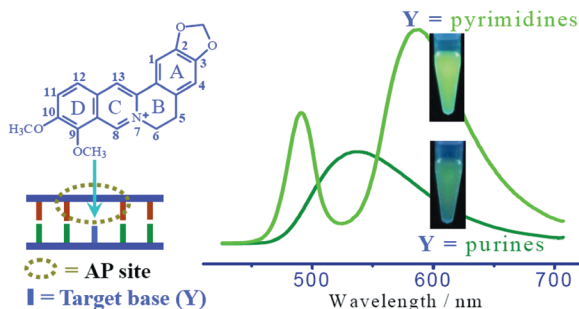
Synthesis and evaluation of novel aza-caged *Garcinia* xanthones

Xiaojin Zhang, Xiang Li, Haopeng Sun,* Zhengyu Jiang, Lei Tao, Yuan Gao, Qinglong Guo* and Qidong You*

The novel aza-caged *Garcinia* xanthonone analogues have been synthesized regioselectively and further evaluated as anti-tumor agents and IKK β inhibitors.



3300

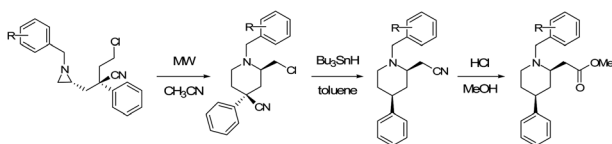


Simultaneous fluorescence light-up and selective multicolor nucleobase recognition based on sequence-dependent strong binding of berberine to DNA abasic site

Fei Wu, Yong Shao,* Kun Ma, Qinghua Cui, Guiying Liu and Shujuan Xu

Sequence-dependent binding of berberine at the DNA abasic site can be used for fluorescence light-up and multicolor nucleobase recognition.

3308

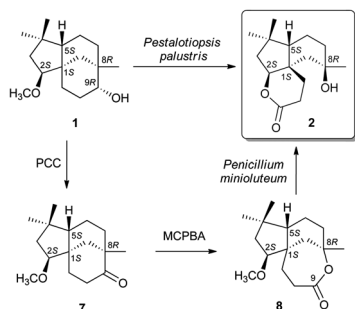


Radical-mediated nitrile translocation as the key step in the stereoselective transformation of 2-(4-chloro-2-cyanobutyl)aziridines to methyl *cis*-(1-arylmethyl-4-phenylpiperidin-2-yl)acetates

Karel Vervisch, Matthias D'hooghe,* Karl W. Törnroos and Norbert De Kimpe*

2-(4-Chloro-2-cyano-2-phenylbutyl)aziridines were transformed stereoselectively into *cis*-2-chloromethyl-4-phenylpiperidine-4-carbonitriles via a microwave-assisted aziridine to piperidine ring expansion, followed by a radical-induced nitrile translocation to afford *cis*-2-cyanomethyl-4-phenylpiperidines.

3315

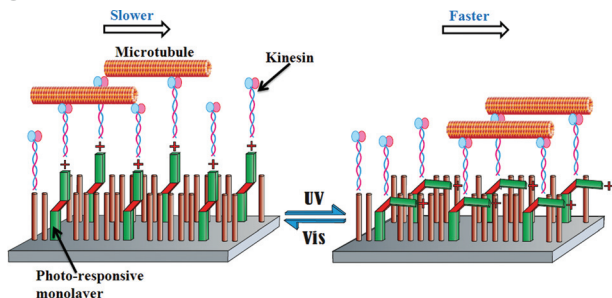


Biotransformation of clovane derivatives. Whole cell fungi mediated domino synthesis of rumphelloclovane A

Giovanni Gontijo de Souza, Thays Silva Oliveira, Jacqueline Aparecida Takahashi, Isidro González Collado, Antonio José Macías-Sánchez* and Rosario Hernández-Galán*

Methoxyclovanol (**1**), is metabolised by *Pestalotiopsis palustris* to yield the natural product rumphelloclovane A (**2**) together with two new clovane derivatives, **5** and **6**. *Penicillium minioluteum* is able to selectively transform lactone **8**, into compound **2** via a domino process.

3321



Dynamic photo-control of kinesin on a photoisomerizable monolayer – hydrolysis rate of ATP and motility of microtubules depending on the terminal group

M. K. Abdul Rahim, Takashi Kamei and Nobuyuki Tamaoki*

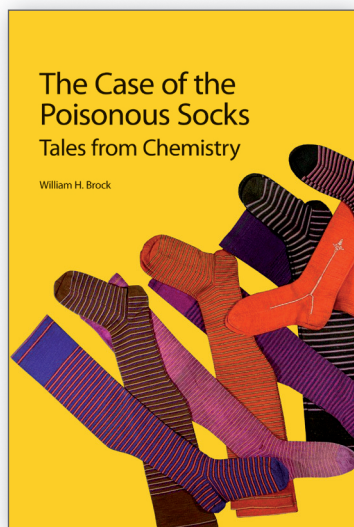
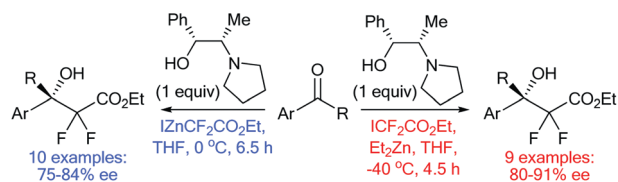
The reversibly and repeatedly altered gliding motility of microtubules driven by kinesin on the photoresponsive monolayer surface is studied.

3332

Enantioselective Reformatsky reaction of ethyl iododifluoroacetate with ketones

Michal Fornalczyk, Kuldip Singh and Alison M. Stuart*

Two approaches have been developed for the enantioselective Reformatsky reaction of ethyl iododifluoroacetate with alkyl aryl ketones to form a quaternary carbon centre.



The Case of the Poisonous Socks

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William H. Brock

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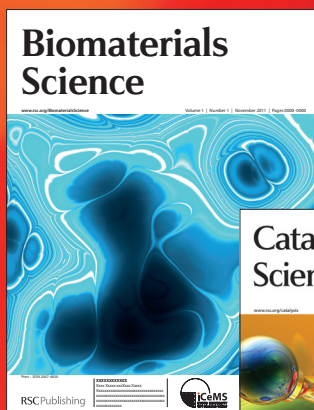
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