

IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (35) 4369-4488 (2005)

In this issue...

Alan Spivey describes a facile method for the preparation of the first solid-supported *N*-methylthiourea reagent and its application to the hydrogenolysis of bicyclic endoperoxides. See pp. 4426 - 4428.



Chemical biology articles published in this journal also appear in the *Chemical Biology Virtual Journal*: www.rsc.org/chembiol



Cover

See Alessio Lodola, Marco Mor, Johannes C. Hermann, Giorgio Tarzia, Daniele Piomelli and Adrian J. Mulholland, page 4399. The cover shows a representation of oleamide hydrolysis by fatty acid amide hydrolase. Image reproduced by permission of Adrian J. Mulholland *et al.* from *Chem. Commun.*, 2005, 4399.

CHEMICAL TECHNOLOGY

T33

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September 2005/Volume 2/Issue 9

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FEATURE ARTICLE

4383

β -1,3-Glucan polysaccharides as novel one-dimensional hosts for DNA/RNA, conjugated polymers and nanoparticles

Kazuo Sakurai, Kazuya Uezu, Munenori Numata, Teruaki Hasegawa, Chun Li, Kenji Kaneko and Seiji Shinkai*

β -1,3-Glucans can form stable complexes with various guests including polynucleotides, conjugate polymers, diacetylene monomers, Au nanoparticles, *etc.* This unique property is quite useful to develop various advanced materials, such as non-toxic carriers and linear Au-nanoarrays.



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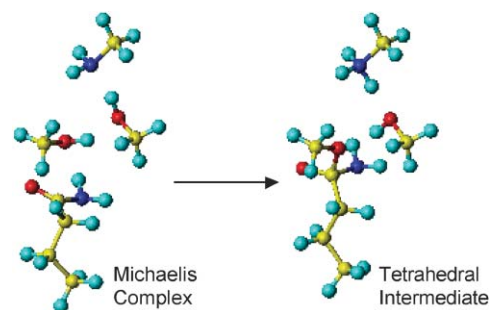
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QM/MM modelling of oleamide hydrolysis in fatty acid amide hydrolase (FAAH) reveals a new mechanism of nucleophile activation

Alessio Lodola, Marco Mor, Johannes C. Hermann, Giorgio Tarzia, Daniele Piomelli and Adrian J. Mulholland*

Fatty acid amide hydrolase (FAAH) is a promising target for the treatment of several central and peripheral nervous system disorders. QM/MM calculations reveal a new mechanism of nucleophile activation, with potentially crucial insights for the design of potent and selective inhibitors.

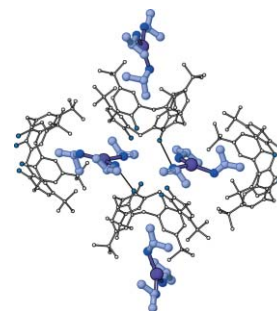


4402

Extending the chemistry of *p*-tert-butylcalix[4]arene with H-bonding and secondary coordination

Philip O. Brown, Konstantin A. Udachin, Gary D. Enright and John A. Ripmeester*

p-tert-Butylcalix[4]arene, together with amines, water and metals, forms novel compounds containing complex clusters and layers.

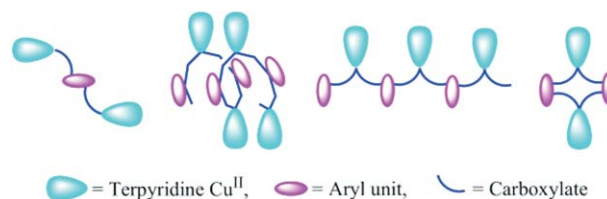


4405

Terpyridine copper^{II}–polycarboxylic acid architectures: formation of dimeric, helical, and cyclic nanostructures and their included-water molecule motifs

Pingshan Wang, Charles N. Moorefield, Matthew Panzer and George R. Newkome*

New crystalline architectures have been prepared employing the propensity of terpyridine–Cu^{II} adducts to form coordination complexes with carboxylate moieties. The crystal structures were analyzed and solved by X-ray diffraction.

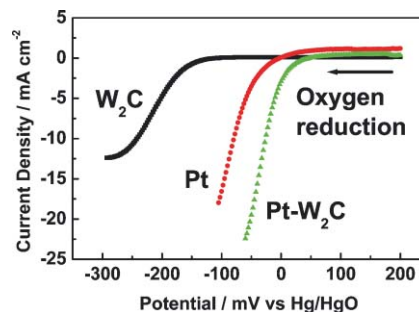


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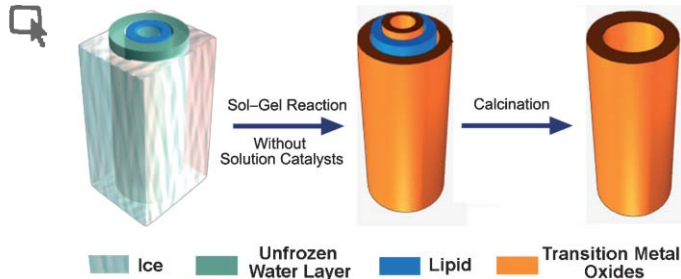
The beneficial effect of the addition of tungsten carbides to Pt catalysts on the oxygen electroreduction

Hui Meng and Pei Kang Shen*

Tungsten carbide nanocrystal modified Pt catalysts have been prepared by an intermittent microwave heating (IMH) method and show an improved activity for oxygen electroreduction in alkaline media.



4411

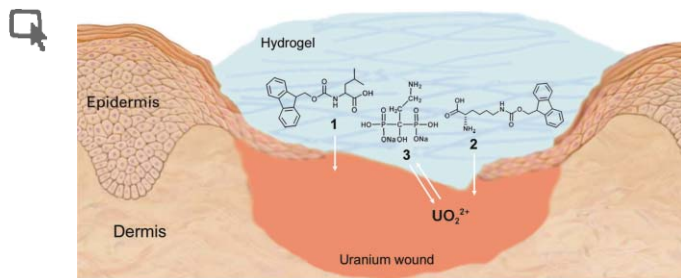


Chemical synthesis of transition metal oxide nanotubes in water using an iced lipid nanotube as a template

Qingmin Ji and Toshimi Shimizu*

An iced lipid nanotube has been employed as a template to perform sol-gel transcription of transition metal alkoxides in the absence of solution catalysts, giving titania, tantalum oxide and vanadium oxide nanotubes in aqueous dispersions.

4414

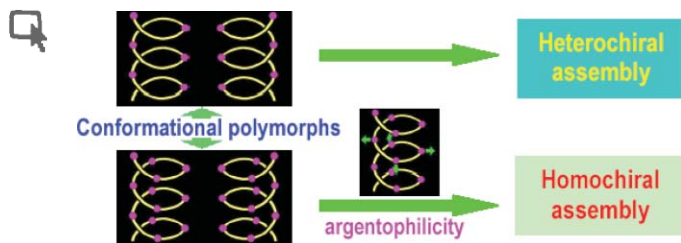


Self-assembly of small molecules affords multifunctional supramolecular hydrogels for topically treating simulated uranium wounds

Zhimou Yang, Keming Xu, Ling Wang, Hongwei Gu, Heng Wei, Mingjie Zhang and Bing Xu*

A multifunctional supramolecular hydrogel reduces the toxicity of uranyl oxide at the wound sites.

4417

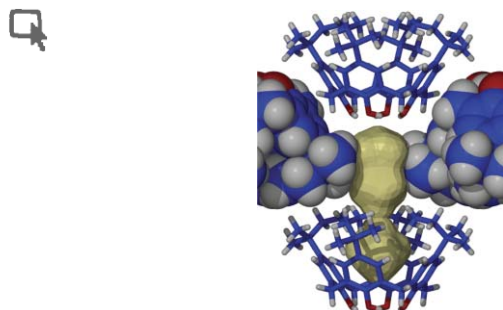


Controlled generation of heterochiral or homochiral coordination polymer: helical conformational polymorphs and argentophilicity-induced spontaneous resolution

Xu-Dong Chen, Miao Du and Thomas C. W. Mak*

In the pair of conformational polymorphs $\{[AgL](CF_3SO_3)\}_\infty$ ($L = 2$ -pyridinyl-3-pyridinylmethanone), one supramolecular isomer contains 2_1 helices of opposite chirality, while the other has homochiral 4_1 helices assembled by inter-chain argentophilic interaction.

4420



Crystal engineering of nonporous organic solids for methane sorption

Praveen K. Thallapally, Trevor B. Wirsig, Leonard J. Barbour* and Jerry L. Atwood*

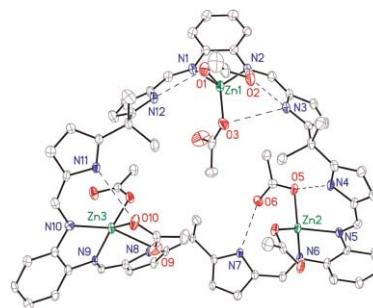
The low density polymorph of the well-known host *p*-*tert*-butylcalix[4]arene absorbs more methane than *p*-*tert*-pentylcalix[4]arene at room temperature and 1 atm pressure, but the order of absorption is reversed at 38 atm with *p*-*tert*-pentylcalix[4]arene absorbing more.

4423

Metal-directed ring-expansion in Schiff-base polypyrrolic macrocycles

Gonzalo Givaja, Alexander J. Blake, Claire Wilson, Martin Schröder and Jason B. Love*

The reaction between a Schiff-base porphyrin analogue and zinc acetate results in the unprecedented formation of a [3 + 3] macrocycle from its [2 + 2] precursor; the [3 + 3] product is stabilised by metal coordination and intramolecular hydrogen-bonding interaction.

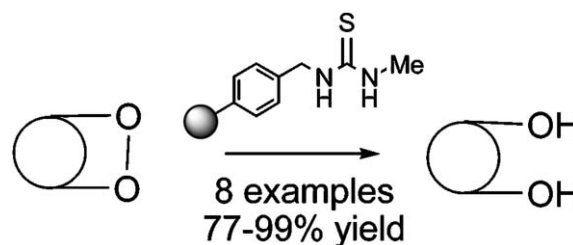


4426

Polystyrene-supported *N*-methylthiourea: a convenient new reagent for the hydrogenolysis of bicyclic endoperoxides

Alan C. Spivey,* Carles Giró Mañas and Inderjit Mann

The single-step preparation of a polystyrene-bound thiourea and its use for the hydrogenolysis of bicyclic endoperoxides is described.

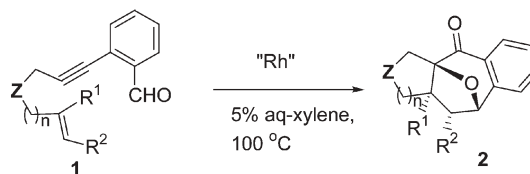


4429

Rhodium-catalyzed tandem cyclization–cycloaddition reactions of enynebenzaldehydes: construction of polycyclic ring systems

Seunghoon Shin, Arun Kumar Gupta, Chul Yun Rhim and Chang Ho Oh*

o-(1,6-Enynyl)benzaldehydes underwent a novel mode of cycloaddition using Rh(I)-precatalyst, via [3 + 2] cycloaddition of presumed Rh-carbenoid dipolar carbonyl ylide intermediate and the utility of this mechanistically intriguing enyne cyclization can be found in a number of polycyclic natural product skeletons.

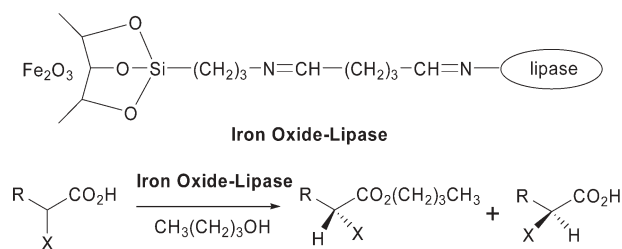


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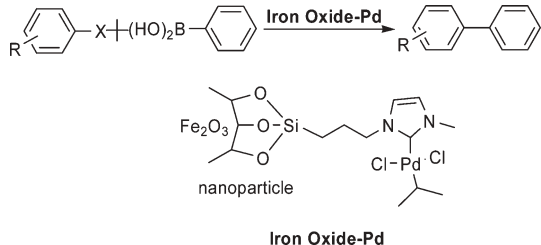
Superparamagnetic nanoparticle-supported enzymatic resolution of racemic carboxylates

Hari M. R. Gardimalla, Deendayal Mandal, Philip D. Stevens, Max Yen and Yong Gao*

Candida rugosa lipase immobilized on maghemite nanoparticles demonstrated high stereoselectivity in kinetic resolution of racemic carboxylates and improved long-term stability over its parent free enzyme.



4435

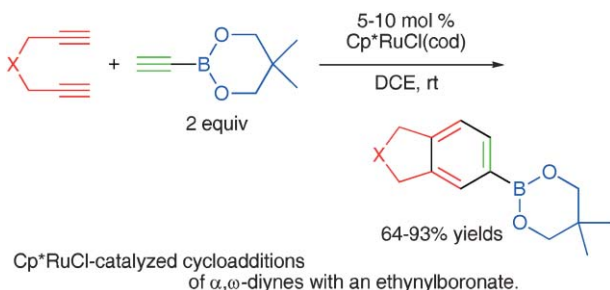


Recycling of homogeneous Pd catalysts using superparamagnetic nanoparticles as novel soluble supports for Suzuki, Heck, and Sonogashira cross-coupling reactions

Philip D. Stevens, Guifeng Li, Jinda Fan, Max Yen and Yong Gao*

Recycling of homogeneous catalysts could be achieved by using magnetic nanoparticles and solid-phase beads, but nanoparticle-supported catalysis proceeded much faster than its counterpart on resins.

4438

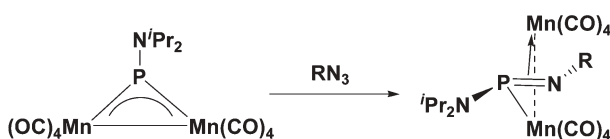


Synthesis of bi- and tricyclic arylboronates via Cp*RuCl-catalyzed cycloaddition of α,ω -diynes with ethynylboronate

Yoshihiko Yamamoto,* Kozo Hattori, Jun-ichi Ishii, Hisao Nishiyama and Kenji Itoh

Polycyclic arylboronates were synthesized *via* Cp*RuCl-catalyzed cycloaddition of α,ω -diynes with 2-ethynyl-5,5-dimethyl-1,3,2-dioxaborinane.

4441

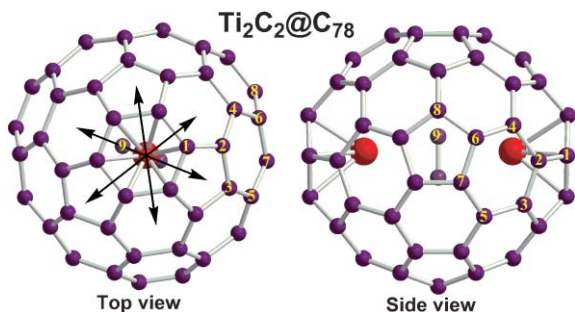


Reactivity of electrophilic μ -phosphinidene complexes with heterocumulenes: formation of the first σ - π -aminophosphimine complexes $[\text{Mn}_2(\text{CO})_8\{\mu\text{-}\eta^1,\eta^2\text{-P}(\text{N}^i\text{Pr}_2)=\text{NR}\}]$ and diazoalkane insertions into metal-phosphorus bonds

Todd W. Graham, Konstantin A. Udachin and Arthur J. Carty*

Phosphinidene complexes $[\text{Mn}_2(\text{CO})_8(\mu\text{-PN}^i\text{Pr}_2)]$ and $[\text{Co}_2(\text{CO})_4(\mu\text{-dppm})(\mu\text{-PNR}_2)]$ react with RN_3 and CR_2N_2 to form complexes with $\mu\text{-}\eta^1,\eta^2\text{-phosphimine}$, $\mu\text{-}\eta^1,\eta^2\text{-phosphaalkene}$ and $\mu\text{-}\eta^1,\eta^2\text{-phosphadiphenylmethylazimine}$ ligands.

4444



Ti₂C₈₀ is more likely a titanium carbide endohedral metallofullerene (Ti₂C₂)@C₇₈

Kai Tan and Xin Lu*

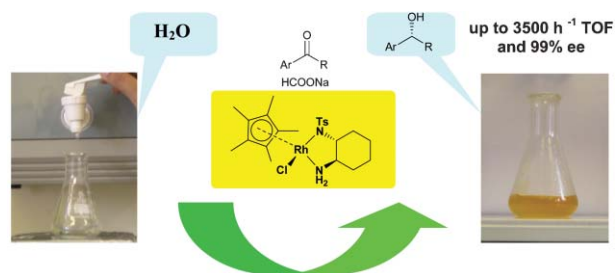
The dimetallofullerene Ti₂C₈₀ does not take the form of Ti₂@C₈₀, but is a titanium carbide endohedral metallofullerene, Ti₂C₂@C₇₈, that has a C₇₈⁶⁻(D_{3h}) cage which follows faithfully the stable closed-shell electronic rule.

4447

A remarkably effective catalyst for the asymmetric transfer hydrogenation of aromatic ketones in water and air

Xiaofeng Wu, Daniele Vinci, Takao Ikariya and Jiangliang Xiao*

A rhodium(III) complex generated *in situ* from $[\text{Cp}^*\text{RhCl}_2]_2$ and (1*R*,2*R*)-*N*-(*p*-toluenesulfonyl)-1,2-cyclohexanediamine (TsCYDN) serves as a remarkably efficient, robust catalyst for the asymmetric transfer hydrogenation of aromatic ketones by HCOONa in water in air, affording chiral alcohols in up to > 99% conversions and 99% ee's.



4450

Efficient nickel catalyst for coupling of acetonitrile with aldehydes

Lei Fan and Oleg V. Ozerov*

A Ni complex of a diarylamido-based PNP ligand is an efficient and robust catalyst for coupling of acetonitrile with aldehydes.

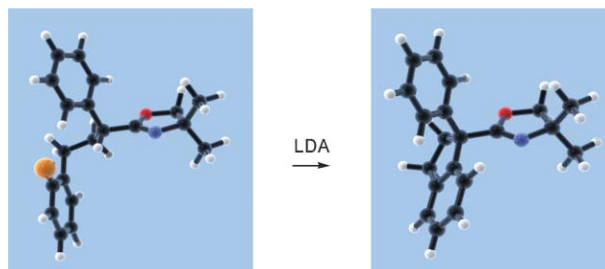


4453

Radical-carbanion cyclo-coupling in armed aromatics: overriding steric hindrance to ring closure

Mark D. Roydhouse and John C. Walton*

ω -(2-Halophenyl)alkyl-2-oxazolines were prepared and reacted *via* base promoted intramolecular coupling of radical with carbanionic centres to yield 1-phenyl-1-oxazolino-indan and -tetralin derivatives containing quaternary C-atoms.

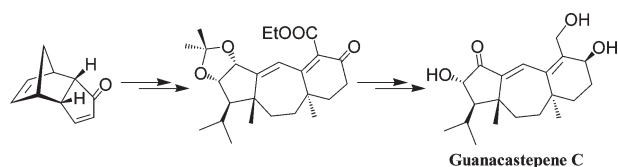


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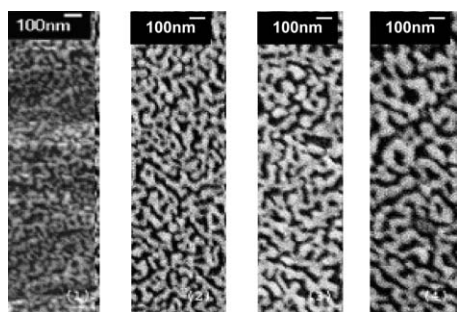
A total synthesis of guanacastepene C

Goverdhan Mehta,* Kotapalli Pallavi and Jayant D. Umarye

A total synthesis of the novel tricyclic diterpene guanacastepene C has been achieved in which a Knoevenagel cyclization has been deployed as a key step to annulate the six-membered C-ring on a previously reported hydroazulene precursor.



4459

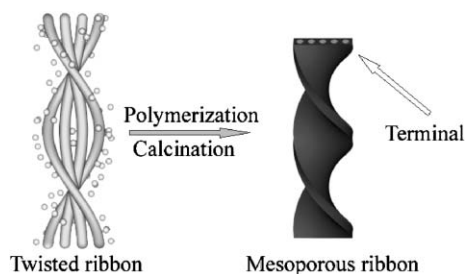


The zwitterion effect in proton exchange membranes as synthesised by polymerisation of bicontinuous microemulsions

Leong Ming Gan,* Pei Yong Chow, Zhaolin Liu, Ming Han and Chai Hoon Quek

Bicontinuous microemulsions consisting of a polymerisable zwitterionic surfactant AIPS and other monomers can be cross-polymerised to form good proton conductive membranes.

4462



Preparation of helical nanostructures using chiral cationic surfactants

Yonggang Yang, Masahiro Suzuki, Sanae Owa, Hirofusa Shirai and Kenji Hanabusa*

Both left-handed helical short nanotubes and mesoporous nanofibers were prepared by sol-gel transcription using a new chiral cationic surfactant.

4465



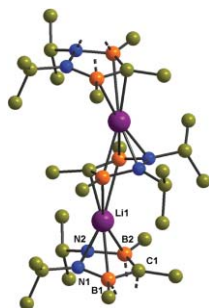
Pictet-Spengler reactions in multiphasic supercritical carbon dioxide/CO₂-expanded liquid media.

In situ generation of carbamates as a strategy for reactions of amines in supercritical carbon dioxide

Joshua R. Dunetz, Rocco P. Ciccolini, Morgan Fröling, Scott M. Paap, Andrew J. Allen, Andrew B. Holmes, Jefferson W. Tester* and Rick L. Danheiser*

Pictet-Spengler cyclizations can be achieved in scCO₂/CO₂-expanded liquid media *via* the *in situ* formation of carbamate derivatives of β-arylethylamines.

4468



μ - η^3 : η^4 -Lithiocene and η^3 : η^3 -zincocene incorporating 1,2-diaza-3,5-diboroly, a cyclopentadienyl analog

Hanh. V. Ly, Taryn. D. Forster, Darren Maley, Masood Parvez and Roland Roesler*

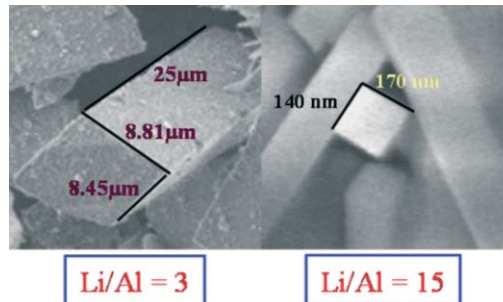
A five-membered N₂B₂C-ring was prepared by ring closure between 1,2-diisopropylhydrazine and 1,1-di(methylchloroboryl)ethane. Deprotonation of the ring produced a heterocyclic cyclopentadienyl analog containing only one carbon atom in the ring. The crystal structures for a polymeric lithiocene and a monomeric zincocene containing this ligand are described.

4471

Surfactant-free hydrothermal synthesis of lithium aluminate microbricks and nanorods from aluminium oxide nanoparticles

Upendra. A. Joshi, Soo Hyun Chung and Jae Sung Lee*

β -LiAlO₂ microbricks and rectangular nanorods have been successfully synthesized from Al₂O₃ nanoparticles by a simple hydrothermal process without any surfactant or template, by simply changing the Li/Al molar ratio.

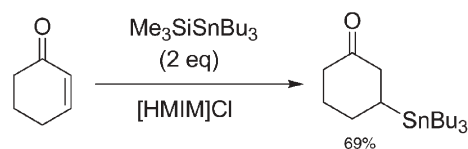


4474

Silylstannations of α,β -unsaturated carbonyl compounds via the generation of Bu₃Sn⁻ in ionic liquids

Steven Dickson, Darrell Dean and Robert. D. Singer*

The tributylstannyl anion, Bu₃Sn⁻, can be generated in imidazolium based ionic liquids from Me₃SiSnBu₃ and reacted with α,β -unsaturated carbonyl compounds to afford 3-tributylstannylated products in good yields.

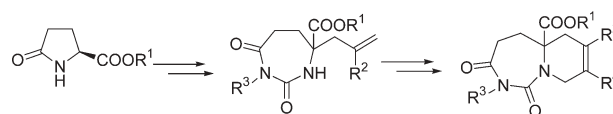


4477

Synthesis of 1,3-dioxo-hexahydropyrido[1,2-c][1,3]diazepine carboxylates, a new bicyclic skeleton formed by ring expansion–RCM methodology

Nicolai Dieltiens, Diederica D. Claeys, Bart Allaert, Francis Verpoort and Christian V. Stevens*

The development of the new hexahydropyridodiazepine skeleton by a sequential ring expansion and ring closing metathesis generates an interesting scaffold for further elaboration for agrochemical and pharmaceutical applications.

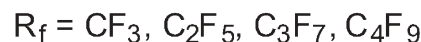


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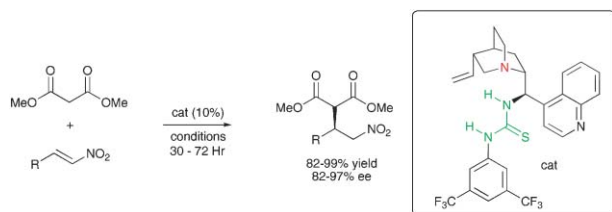
The synthesis of tris(perfluoroalkyl)phosphines

Makeba B. Murphy-Jolly, Lesley C. Lewis and Andrew J. M. Caffyn*

Tris(perfluoroalkyl)phosphines can be synthesised by the nucleophile mediated reaction of perfluoroalkyltrimethylsilanes with P(OPh)₃; the method can be extended to diphosphines. These phosphines are of interest as tunable alternatives to the carbon monoxide ligand.



4481

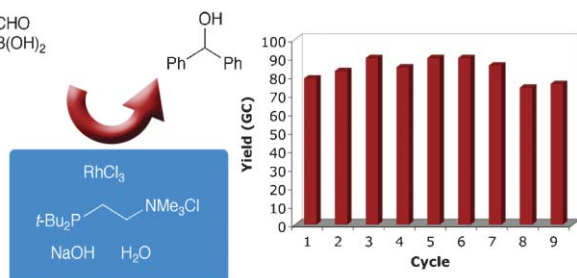


Enantioselective organocatalytic Michael addition of malonate esters to nitro olefins using bifunctional cinchonine derivatives

Jinxing Ye, Darren J. Dixon* and Peter S. Hynes

A novel asymmetric Lewis base–Brønsted acid bifunctional organic catalyst promotes high reactivity and enantioselectivity in the Michael addition of malonate esters to nitro olefins.

4484



t-Bu-Amphos– $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$: a highly recyclable catalyst system for the cross-coupling of aldehydes and aryl- and alkenylboronic acids in aqueous solvents

Rongcai Huang and Kevin H. Shaughnessy*

The combination of *t*-Bu-Amphos and $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ gave the first highly recyclable catalyst for the coupling of aryl- and vinylboronic acids with aldehydes in aqueous solvents.

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