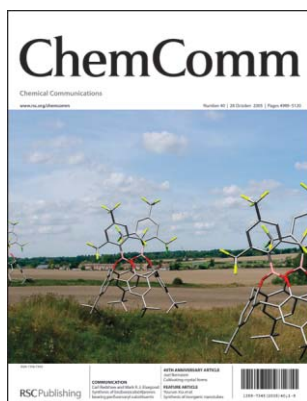


IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (40) 4989-5120 (2005)



Cover

See Carl Redshaw *et al.*, page 5056. Calixarene chemistry is flourishing in East Anglia: here perfluoroaryl bis(bora)calix[4]arenes align in pylon-type fashion against a typical Norfolk landscape. Image reproduced by permission of Carl Redshaw and Mark R. J. Elsegood from *Chem. Commun.*, 2005, 5056–5058.



Inside cover

See Shuxia Liu *et al.*, page 5023. Hexadecavanadate clusters and a 3D nickel complex have combined to form a unique organic–inorganic hybrid material. Image reproduced by permission of Shuxia Liu, Linhua Xie, Bo Gao, Chundan Zhang, Chunyan Sun, Dehui Li and Zhongmin Su, from *Chem. Commun.*, 2005, 5023–5025.

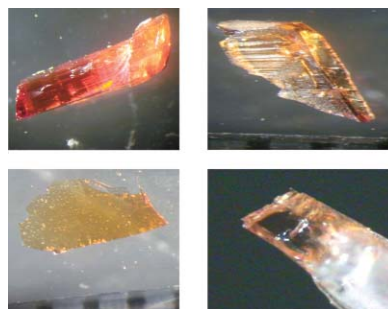
40TH ANNIVERSARY ARTICLE

5007

Cultivating crystal forms

Joel Bernstein

A plethora of new crystal forms – polymorphs, solvates and co-crystals – are being prepared both by design, using a variety of new and newly revived techniques, as well as being discovered by serendipity. Pictured are crystals of four polymorphs of benzidine used to determine the previously unreported crystal structures.



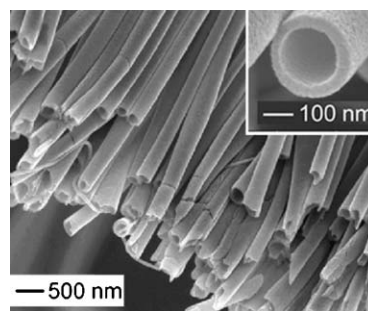
FEATURE ARTICLE

5013

Some recent developments in the chemical synthesis of inorganic nanotubes

Yujie Xiong, Brian T. Mayers and Younan Xia*

Inorganic nanotubes have been a subject of intensive research in the past decade. We recently developed a number of synthetic strategies for generating nanotubes from inorganic materials that do not have a layered structure. It is the intention of this contribution to provide a brief account of these research activities.



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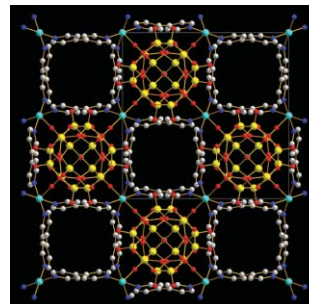
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5023

An organic–inorganic hybrid material constructed from a three-dimensional coordination complex cationic framework and entrapped hexadecavanadate clusters

Shuxia Liu,* Linhua Xie, Bo Gao, Chundan Zhang, Chunyan Sun, Dehui Li and Zhongmin Su

A unique organic–inorganic hybrid compound has been separated under hydrothermal conditions, which is constructed from a three-dimensional second metal–organic subunit and entrapped hexadecavanadate clusters.

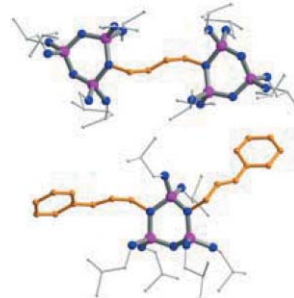


5026

Connecting cyclophosphazenes *via* ring N-centres with covalent linkers

Mark A. Benson and Alexander Steiner*

Two cyclotriphosphazene rings can be covalently linked *via* ring N-centres by a 2-butene-1,4-diyl unit and *vice versa* a cyclotriphosphazene molecule is able to bridge two cinnamyl groups *via* two ring N-centres yielding in either case dicationic assemblies which offers a route to novel polycations.

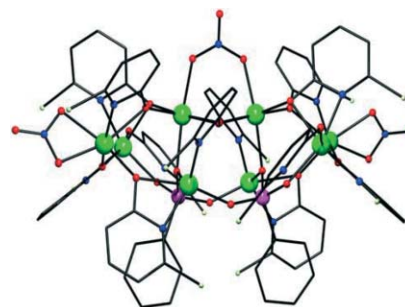


5029

Slow relaxation of magnetisation in an octanuclear cobalt(II) phosphonate cage complex

Stuart J. Langley, Madeleine Helliwell, Roberta Sessoli,* Patrick Rosa, Wolfgang Wernsdorfer* and Richard E. P. Winpenny*

Studies of a new $\{Co_8\}$ cluster show an apparently high energy barrier for loss of magnetisation if it was a “single molecule magnet”, but unusual magnetic hysteresis behaviour.

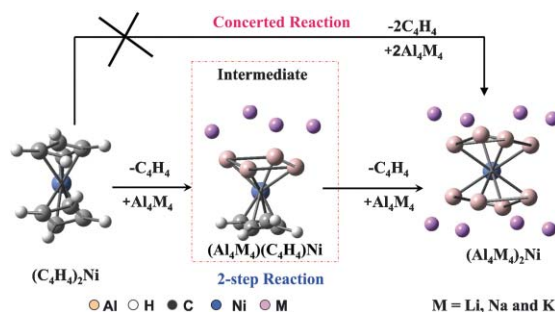


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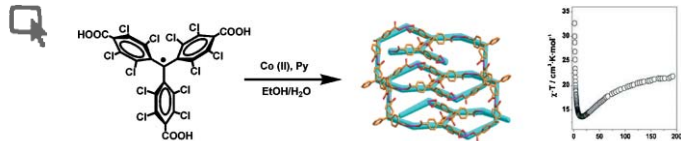
New examples of metalloaromatic Al-clusters: $(Al_4M_4)Fe(CO)_3$ ($M = Li, Na$ and K) and $(Al_4M_4)_2Ni$: rationalization for possible synthesis

Ayan Datta and Swapan K. Pati*

Quantum chemical calculations offer the possibility of realizing new all-metal coordination sandwich complexes of Al_4M_4 through thermodynamically favourable step-wise reactions.



5035

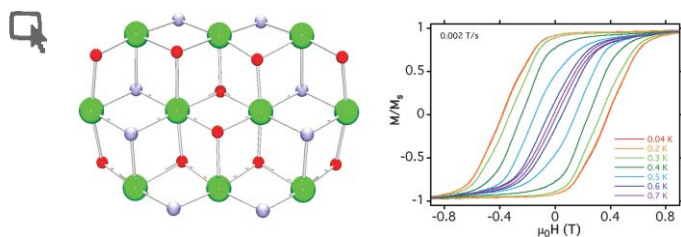


Coexistence of ferro- and antiferromagnetic interactions in a metal–organic radical-based (6,3)-helical network with large channels

Daniel Maspoch, Neus Domingo, Daniel Ruiz-Molina,* Klaus Wurst, Joan-Manel Hernández, Gavin Vaughan, Concepció Rovira, Francesc Lloret, Javier Tejada and Jaume Veciana*

A metal–organic open-framework that exhibits an unusual topology, 1-D nanochannels and mixed ferro- and antiferromagnetic interactions between Co(II) and radical units.

5038

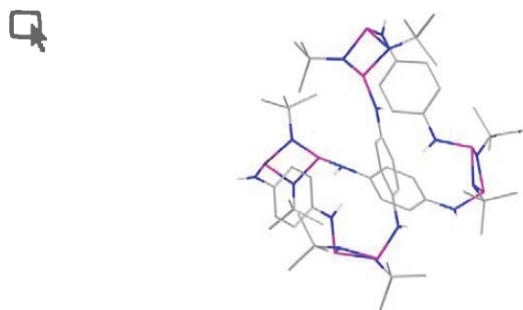


Synthesis, structure and magnetic properties of a decametallc Ni single-molecule magnet

Guillem Aromí, Simon Parsons, Wolfgang Wernsdorfer, Euan K. Brechin* and Eric J. L. McInnes*

Ferromagnetic exchange between the ten Ni^{2+} ions in the complex $[\text{Ni}_{10}(\text{tmp})_2(\text{N}_3)_8(\text{acac})_6(\text{MeOH})_6]$ leads to a spin ground state of $S = 10$. Single crystal M vs. H studies reveal the temperature and sweep rate dependent hysteresis loops expected for a single-molecule magnet.

5041

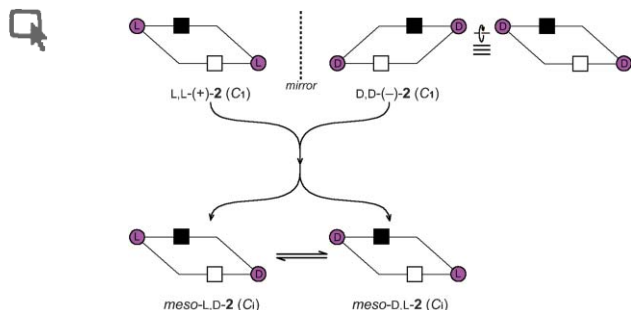


The folded, tetrameric phosph(III)azane macrocycle $[\{\text{P}(\mu\text{-N}^t\text{Bu})\}_2\{1,4\text{-(NH)}_2\text{C}_6\text{H}_4\}]_4$

Fay Dodds, Felipe García, Richard A. Kowenicki, Mary McPartlin, Lucía Riera, Alexander Steiner and Dominic S. Wright*

The tetrameric macrocycle $[\{\text{P}(\mu\text{-N}^t\text{Bu})\}_2\{1,4\text{-(NH)}_2\text{C}_6\text{H}_4\}]_4$ has an unusual folded conformation in the solid state, containing a roughly tetrahedral arrangement of *endo* N–H groups for the potential coordination of anions.

5044



Stereoisomerism in polyoxometalates: structural and spectroscopic studies of bis(malate)-functionalized cluster systems

Xikui Fang, Travis M. Anderson, Yu Hou and Craig L. Hill*

Desymmetrization of centrosymmetric polytungstate clusters by introduction of multiple chiral organic ligands allows the isolation of enantiopure isomers; their interconversion to *meso* diastereomers is demonstrated.

5047

Triplet ground state ($S = 1$) pegylated bis(aminoxyl) diradical: synthesis and the effect of water on magnetic properties

Gaëlle Spagnol, Kouichi Shiraishi, Suchada Rajca and Andrzej Rajca*

The synthesis and magnetic characterization of pegylated bis(aminoxyl) diradical with an $S = 1$ ground state are presented, revealing water-induced changes in the molecular conformation and magnetic properties.

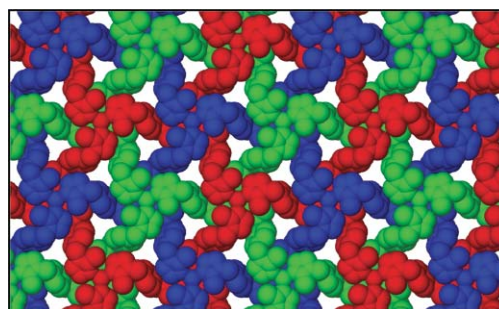


5050

Borromean sheets assembled by self-supporting argentophilic interactions

Liliana Dobrzańska, Helgard G. Raubenheimer and Leonard J. Barbour*

Attractive and cooperative $\text{Ag}\cdots\text{Ag}$ interactions effect the self-assembly of a two-dimensional coordination polymeric structure possessing the Borromean entanglement.

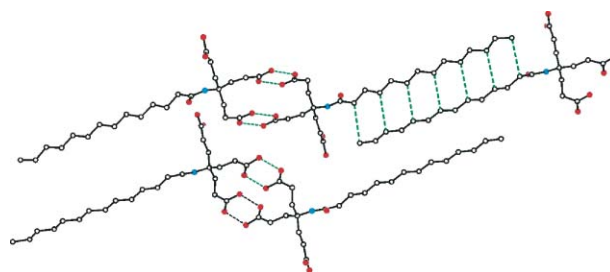


5053

Homologous, long-chain alkyl dendrons form homologous thin films on silver oxide surfaces

André A. Williams, B. Scott Day, Brett L. Kite, Melinda K. McPherson, Carla Slebodnick, John R. Morris* and Richard D. Gandour*

As suggested by X-ray crystal structures, homologous, long-chain alkyl dendrons with three carboxyl groups form thin films on silver oxide surfaces, which give reflection-absorption infrared spectra that show a linear increase in intensities of methylene C–H stretching absorptions.

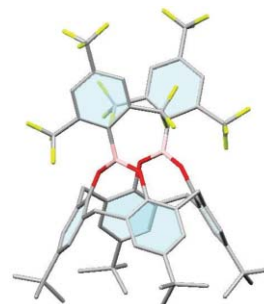


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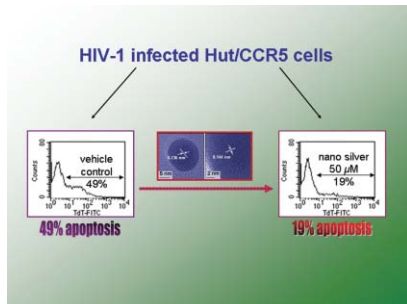
Synthesis of bis(bora)calix[4]arenes bearing perfluoroaryl substituents

Carl Redshaw* and Mark R. J. Elsegood

New routes to perfluoroaryl bis(bora)calix[4]arenes are described; crystallographic studies reveal constrained conformations and reveal a rare example of Cl–F exchange at boron.



5059

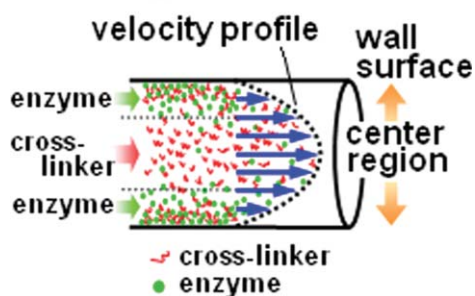


Silver nanoparticles fabricated in Hepes buffer exhibit cytoprotective activities toward HIV-1 infected cells

Raymond Wai-Yin Sun, Rong Chen, Nancy P.-Y. Chung, Chi-Ming Ho, Chen-Lung Steve Lin* and Chi-Ming Che*

Silver nanoparticles fabricated in Hepes buffer exhibit potent cytoprotective and post-infected anti-HIV-1 activities toward Hut/CCR5 cells.

5062

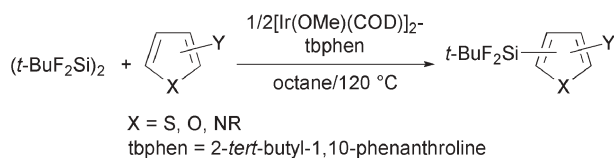


Immobilization of enzymes on a microchannel surface through cross-linking polymerization

Takeshi Honda, Masaya Miyazaki,* Hiroyuki Nakamura and Hideaki Maeda*

We have developed a novel and facile method for the preparation of an enzyme-immobilized microreactor. Enzymes were immobilized as an enzyme-polymer membrane formed on the inner wall of the microchannel by a cross-linking polymerization method.

5065

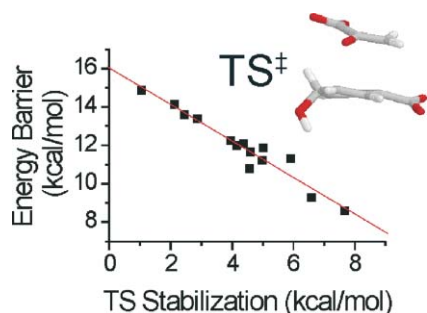


Regioselective aromatic C–H silylation of five-membered heteroarenes with fluorodisilanes catalyzed by iridium(I) complexes

Tatsuo Ishiyama,* Kazuaki Sato, Yukihiro Nishio, Takeaki Saiki and Norio Miyaura*

The aromatic C–H silylation of five-membered heteroarenes with 1,2-di-*tert*-butyl-1,1,2,2-tetrafluorodisilane regioselectively proceeded at 120 °C in octane in the presence of a catalytic amount of iridium(I) complexes generated from $1/2[\text{Ir}(\text{OMe})(\text{COD})]_2$ and 2-*tert*-butyl-1,10-phenanthroline.

5068



Multiple high-level QM/MM reaction paths demonstrate transition-state stabilization in chorismate mutase: correlation of barrier height with transition-state stabilization

Frederik Claeysens, Kara E. Ranaghan, Frederick R. Manby, Jeremy N. Harvey and Adrian J. Mulholland*

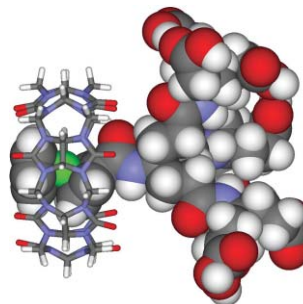
Chorismate mutase is at the centre of current debates on enzyme catalysis. DFT/MM barriers agree with experiment and show significant TS stabilization by the enzyme.

5071

Binding interactions between the host cucurbit[7]uril and dendrimer guests containing a single ferrocenyl residue

David Sobransingh and Angel E. Kaifer*

The thermodynamic stability of the inclusion complexes formed between the host cucurbit[7]uril and dendrimer guests containing a single ferrocenyl residue increases with molecular weight.

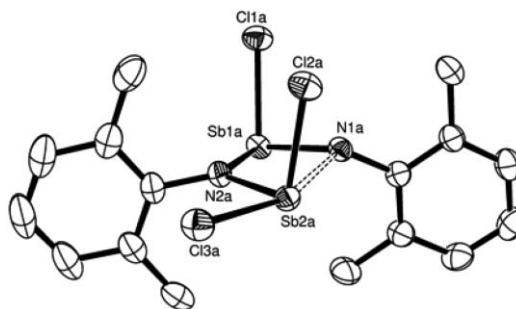


5074

Identification of new N–Sb topologies: understanding the sequential dehydrochloride coupling of primary amines and trichloropnictines

Neil Burford,* Ezra Edelstein, Jeff C. Landry, Michael J. Ferguson and Robert McDonald

The subtle steric strain imposed by substituents on N–Sb frameworks has enabled identification of an amine–stibine adduct, a bisamine–stibine adduct, the first acyclic dipnictadiazane and the first six-membered cyclotristibatriazane.

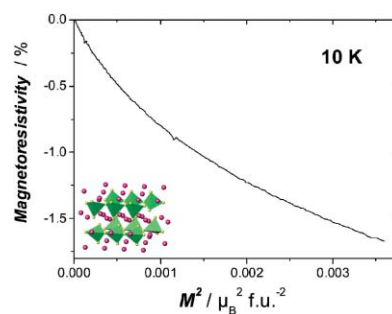


5077

Negative magnetoresistance in Ba₂CoS₃

Thomas Baikie, Vincent Hardy, Antoine Maignan and M Grazia Francesconi*

Ba₂CoS₃ is the first 2-1-3 sulfide to show conducting-like behaviour and a rare example of a one-dimensional sulfide with negative magnetoresistance.



5080

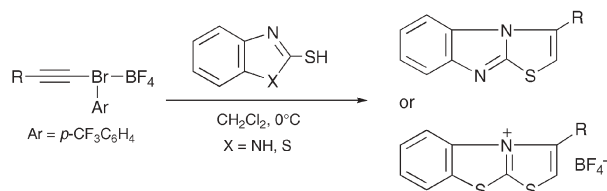
Trialkylsulfonium dicyanamides - a new family of ionic liquids with very low viscosities

Dirk Gerhard, Samim Cenk Alpaslan, Heiner Jakob Gores, Marc Uerdingen and Peter Wasserscheid*

Trialkylsulfonium dicyanamides show surprisingly low viscosities down to $-20\text{ }^{\circ}\text{C}$ and are therefore highly interesting liquid materials for separation processes and electrolyte applications at low temperatures.



5083

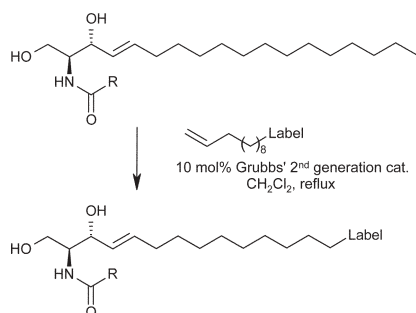


Domino Michael addition-carbene rearrangement-cyclization reaction of 1-alkynyl(aryl)- λ^3 -bromanes with 2-mercapto-1,3-benzazoles

Masahito Ochiai* and Norihiro Tada

Exposure of 1-alkynyl[*p*-(trifluoromethyl)phenyl]-(tetrafluoroborato)- λ^3 -bromanes to 2-mercaptobenzimidazole or benzothiazole in CH_2Cl_2 at 0°C under argon resulted in a domino Michael addition-carbene rearrangement-cyclization to give directly tricyclic heterocycles in high yields, whereas the reaction with 2-mercaptobenzoxazole gave 1-alkynyl sulfides.

5086

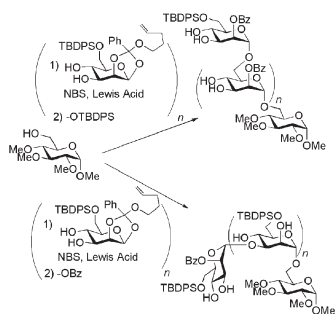


One-step labelling of sphingolipids via a scrambling cross-metathesis reaction

Peter Nussbaumer,* Peter Ettmayer, Carsten Peters, Daniela Rosenbeiger and Klemens Högenauer

The alkyl chain in the backbone of sphingosine derivatives can be exchanged with functionalised (labelled) side chains in a single step under cross-metathesis reaction conditions.

5088

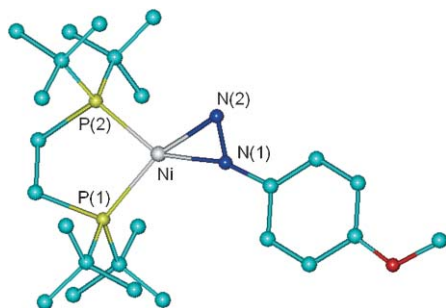


Iterative, orthogonal strategy for oligosaccharide synthesis based on the regioselective glycosylation of polyol acceptors with partially unprotected *n*-pentenyl-orthoesters: further evidence for reciprocal donor acceptor selectivity (RDAS)

J. Cristóbal López,* Attila Agocs, Clara Uriel, Ana M. Gómez and Bert Fraser-Reid

An efficient iterative, orthogonal protocol based on the regioselective glycosyl coupling of *D*-mannose polyols with, partially unprotected, *n*-pentenyl orthoester donors permits the synthesis of linear and branched oligosaccharides.

5091



Synthesis and characterization of side-bound aryldiazo and end-bound nitrosyl complexes of nickel

Vlad M. Iluc, Alexander J. M. Miller and Gregory L. Hillhouse*

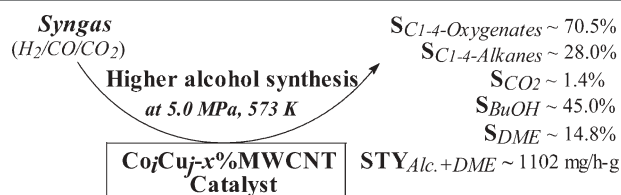
Reaction of an aryldiazonium salt with a $\text{Ni}(0)$ phosphine complex gives an unusual side-bound aryldiazo adduct, but nitrosonium adds in an end-on fashion.

5094

Carbon nanotube-promoted Co–Cu catalyst for highly efficient synthesis of higher alcohols from syngas

Hong-Bin Zhang,* Xin Dong, Guo-Dong Lin, Xue-Lian Liang and Hai-Yan Li

A carbon-nanotube-promoted Co_xCu_y catalyst achieves highly selective formation of BuOH and DME from syngas, and demonstrates great potential in commercial use for higher alcohol synthesis.

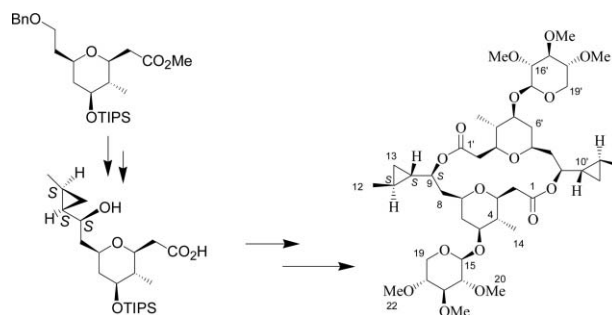


5097

Total synthesis of a diastereomer of the marine natural product clavosolide A

Conor S Barry, Nick Bushby, Jonathan P. H. Charmant, Jon D. Elsworth, John R. Harding and Christine L. Willis*

The total synthesis of the reported structure of the sponge metabolite clavosolide A is described and the target molecule was found to be a diastereomer of the natural product.

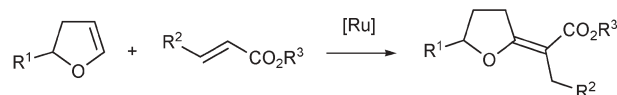


5100

Synthesis of 2-alkylidenetetrahydrofurans by Ru-catalyzed regio- and stereoselective codimerization of dihydrofurans with α,β -unsaturated esters

Hiroshi Tsujita, Yasuyuki Ura, Kenji Wada, Teruyuki Kondo and Take-aki Mitsudo*

2-(1-Alkoxy carbonyl)alkylidenetetrahydrofurans were readily synthesized by the codimerization of 2,3- or 2,5-dihydrofurans with α,β -unsaturated esters using a zerovalent Ru catalyst, $\text{Ru}(\text{cod})(\text{cot})$ [cod = 1,5-cyclooctadiene, cot = 1,3,5-cyclooctatriene], with high regio- and stereoselectivity.

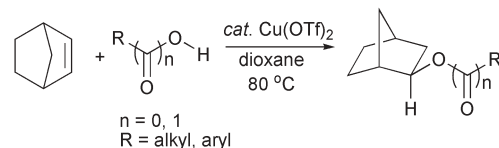


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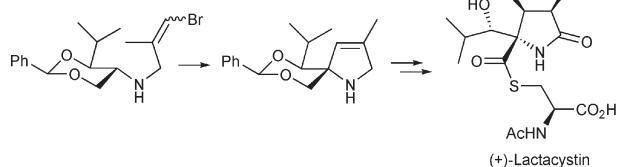
Copper(II)-catalysed addition of O–H bonds to norbornene

Jason G. Taylor, Neil Whittall and King Kuok (Mimi) Hii*

$\text{Cu}(\text{OTf})_2$ is an inexpensive, air- and moisture-stable catalyst for the O–H addition of aliphatic and aromatic acids and alcohols to norbornene.



5106

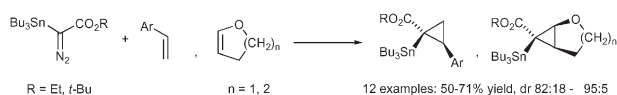


A formal synthesis of (+)-lactacystin

Duncan J. Wardrop* and Edward G. Bowen

A formal synthesis of the proteasome inhibitor, (+)-lactacystin is reported. The key step in this endeavor involves the generation of the C-5 quaternary stereocenter *via* intramolecular C–H insertion of an alkylidenecarbene intermediate.

5109

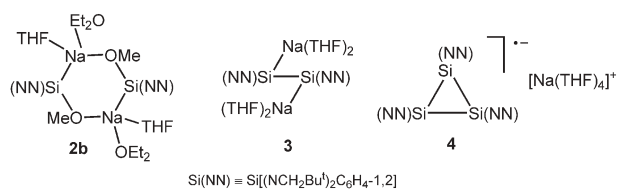


Stannyl cyclopropanes by diastereoselective cyclopropanations with (tributylstannyl)-diazoacetate esters catalyzed by Cu(I) *N*-heterocyclic carbene

Robert E. Gawley* and Sanjay Narayan

Stannyl cyclopropanes having a quaternary tin-bearing carbon atom are made using a NHC copper catalyst in good yields and excellent diastereoselectivity, with no competing dimerization of carbene.

5112



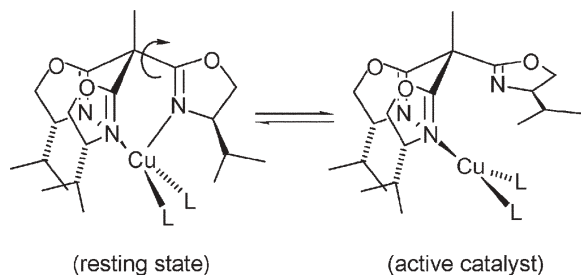
Crystalline Na–Si(NN) derivatives

[Si(NN) = Si{(NCH₂tBu)₂C₆H₄-1,2}]: the silylenoid [Si(NN)OMe][−], the dianion [(NN)Si–Si(NN)]^{2−}, and the radical anion *c*-[Si(NN)]₃[−]

Floria Antolini, Barbara Gehrhus,* Peter B. Hitchcock and Michael F. Lappert*

Reactions of the silylene Si[(NCH₂tBu)₂C₆H₄-1,2] with NaOMe, excess Na or 1/3 Na yield the X-ray-characterised crystalline compounds **2b**, **3** and **4**.

5115



Exploiting C₃-symmetry in the dynamic coordination of a chiral trisoxazoline to copper(II): improved enantioselectivity, and catalyst stability in asymmetric lewis acid catalysis

Carole Foltz, Björn Stecker, Guido Marconi, Stéphane Bellemin-Lapponnaz,* Hubert Wadepohl and Lutz H. Gade*

C₃-symmetric trisoxazolines form highly efficient enantioselective Cu^{II} Lewis acid catalysts which are based on the concept of *stereoelectronic hemilability*.


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