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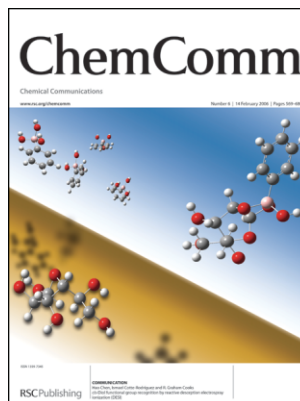
IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (6) 569–680 (2006)



Cover

See Zongbin Zhao, Jiangying Qu, Jieshan Qiu, Xuzhen Wang and Zhiyu Wang, page 594. From carbon nanotubes to aligned microtubes: *in situ* self-assembly in the presence of oxidizing agent. Image reproduced by permission of Jieshan Qiu *et al.*, from *Chem. Commun.*, 2006, 594.



Inside cover

See Hao Chen, Ismael Cotte-Rodríguez and R. Graham Cooks, page 597. Reactant benzenboronate ions $\text{PhB}(\text{OH})_3^-$ impact solid glucose and release covalently-bound cyclic boronate ions in a heterogeneous ion/molecule reaction which occurs in air. Image created by Ouyang Zheng and Hao Chen and reproduced by permission of R. Graham Cooks *et al.*, from *Chem. Commun.*, 2006, 597.

CHEMICAL TECHNOLOGY

T5

Chemical Technology highlights the latest applications and technological aspects of research across the chemical sciences.

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February 2006/Volume 3/Issue 2

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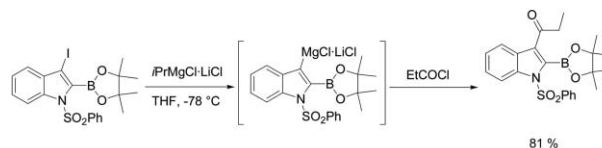
FEATURE ARTICLE

583

Functionalized magnesium organometallics as versatile intermediates for the synthesis of polyfunctional heterocycles

Hiriyakkanavar Ila, Oliver Baron, Andreas J. Wagner and Paul Knoche¹*

A broad range of polyfunctional magnesium organometallic intermediates can be readily prepared by a I/Mg - or a Br/Mg -exchange reaction. A variety of functional groups such as an ester, nitrile, iodide, imine and even sensitive groups like nitro, hydroxyl and boronic ester can be tolerated in these organomagnesium compounds. The scope and limitations of this method as well as recent synthetic applications will be reviewed.



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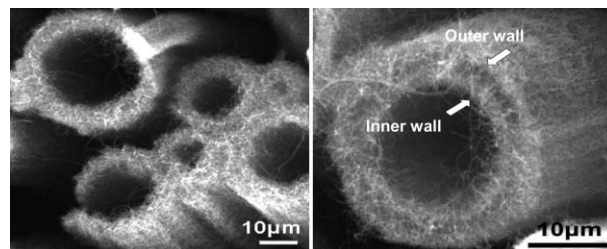
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Water-assisted fabrication of aligned micro-sized carbon tubes made of self-assembled multi-wall carbon nanotubes

Zongbin Zhao, Jiangying Qu, Jieshan Qiu,* Xuzhen Wang and Zhiyu Wang

Aligned micro-sized carbon tubes have been successfully synthesized on silicon substrate by pyrolysis of cyclohexane/ferrocene in the presence of water, a spectacular feature of which is that the multi-wall carbon nanotubes formed *in situ* act as the basic building blocks for the construction of micro-tubes *via* a “multi-scale” self-assembly process.

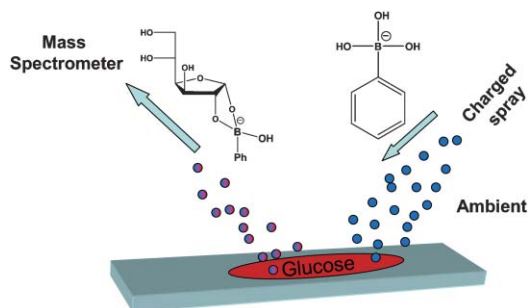


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***cis*-Diol functional group recognition by reactive desorption electrospray ionization (DESI)**

Hao Chen, Ismael Cotte-Rodríguez and R. Graham Cooks*

Heterogeneous reactions at a solution/solid interface are utilized in an ambient mass spectrometry experiment to recognize the *cis*-diol functionality by its selective complexation reaction to form a cyclic boronate.

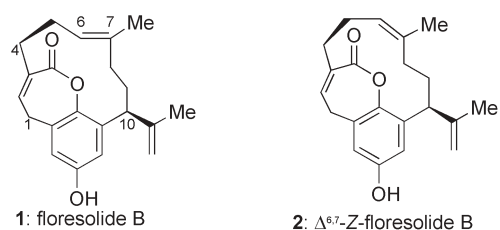


600

Total synthesis of floresolide B and $\Delta^{6,7}$ -Z-floresolide B

K. C. Nicolaou* and Hao Xu

The total syntheses of the cytotoxic marine natural product floresolide B (**1**) and its $\Delta^{6,7}$ -Z isomer (**2**) have been achieved through an olefin metathesis-based strategy.

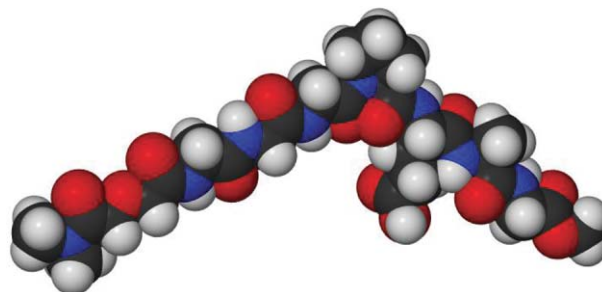


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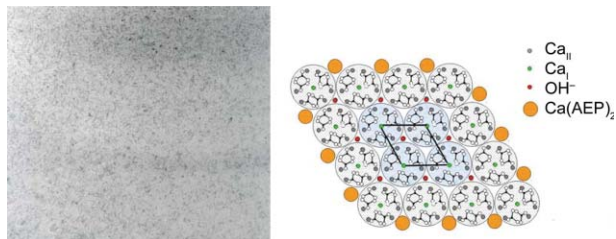
Chloride ion efflux from liposomes is controlled by sidechains in a channel-forming heptapeptide

Lei You, Riccardo Ferdani and George W. Gokel*

A suite of amphiphilic heptapeptides incorporating a glutamic acid derivative on the C-terminal side of a (Gly)₃Pro sequence gives dramatically lower chloride ion release from liposomes when present in free carboxyl form rather than as an ester or amide.



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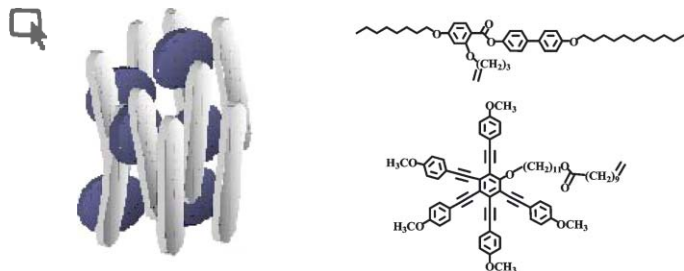


Colloidal and monocrystalline Ln³⁺ doped apatite calcium phosphate as biocompatible fluorescent probes

A. Lebugle, F. Pellé, C. Charvillat, I. Rousselot and J. Y. Chane-Ching*

Ultrafine monocrystalline calcium phosphate nanophosphors displaying fluorescence under visible excitation are proposed for utilisation as biocompatible biological probes.

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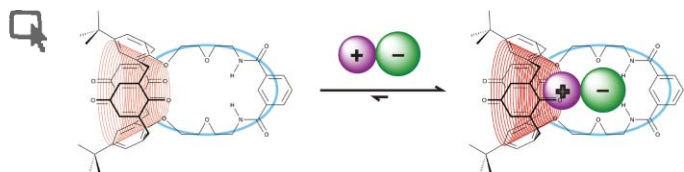


Completely miscible disc and rod shaped molecules in the nematic phase

Daniela Apreutesei and Georg H. Mehl*

The design of rod and disc shaped molecules which mix completely in the nematic phase has been a long standing issue in LC research. The use of materials with relatively similar melting points, containing long alkyl chains is a solution to this problem.

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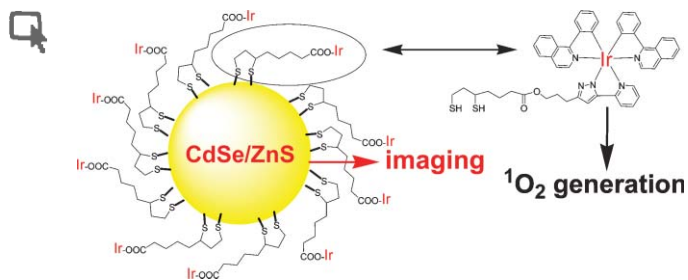


Cooperative AND receptor for ion-pairs

Michael D. Lankshear, Andrew R. Cowley and Paul D. Beer*

A new calix[4]diquinone species has been found to bind simple ion-pair systems strongly where no discernible affinity for either of the free ions is observed.

615



Iridium-complex modified CdSe/ZnS quantum dots; a conceptual design for bifunctionality toward imaging and photosensitization

Jia-Ming Hsieh, Mei-Lin Ho, Pei-Wen Wu, Pi-Tai Chou,* Tsai-Tsung Tsai and Yun Chi*

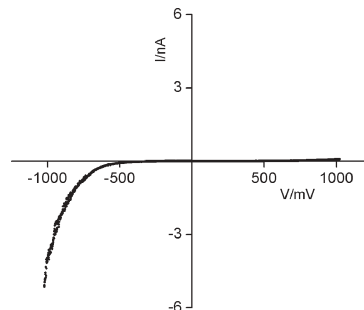
Energy transfer between QDs and Ir-complex is negligible for the Ir-complex functionalized CdSe/ZnS QDs. The central QDs play a key role in imaging, while the Ir-complex acts as a sensitizer to produce singlet oxygen. This conceptual design presents a novel scheme in both bio-imaging and photodynamic therapy.

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Organic rectifying junctions fabricated by ionic coupling

Geoffrey J. Ashwell,* Jonathan Ewington and Benjamin J. Robinson

Ionically-assembled layers of cationic acceptors and anionic donors exhibit asymmetric $I-V$ characteristics with a rectification ratio of 60–100 at ± 1 V, the highest to date for an ultra-thin organic rectifying junction.



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A molecular pinwheel multicopper(I) cluster, $[(L^{S-})_6Cu_{13}(S^{2-})_2]^{3+}$ with μ_4 -sulfido, μ_3 -thiolato and nitrogen ligands

Yunho Lee, Amy A. Narducci Sarjeant and Kenneth D. Karlin*

The cluster $[(L^{S-})_6Cu_{13}(S^{2-})_2]^{3+}$ can be generated from a copper(I) complex with a N_2S thiol ligand; its X-ray structure includes a μ_4 -sulfido Cu_4S core reminiscent of nitrous oxide reductase.

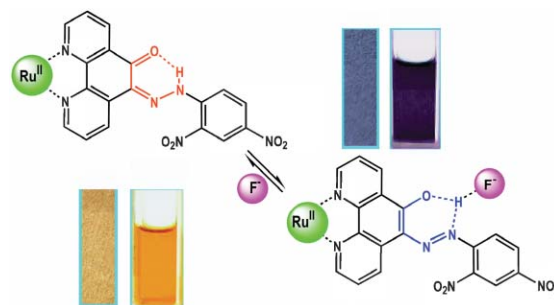


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Naked-eye detection of fluoride ion in water: a remarkably selective easy-to-prepare test paper

Zhi-hua Lin, Sheng-ju Ou, Chun-ying Duan,* Bing-guang Zhang and Zhi-ping Bai*

A test paper for high-selectively detecting F^- in natural aqueous environments without any spectroscopic instrumentation was achieved by using Ru-bipy based quinonehydrazone as a chromo- and fluorogenic hybrid chemosensor.

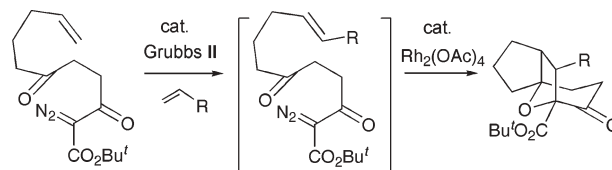


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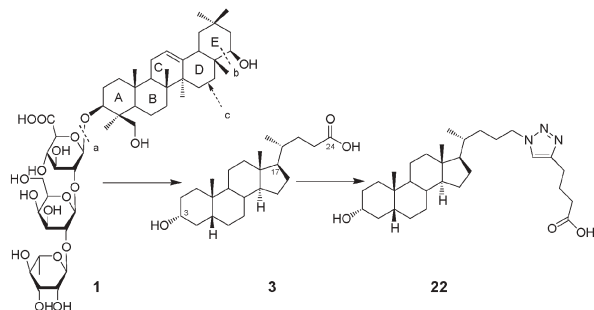
One-pot cross-metathesis/tandem carbonyl ylide formation–intramolecular cycloaddition of an unsaturated 2-diazo-3,6-diketoester

David M. Hodgson,* Deepshikha Angrish and Agnès H. Labande

Rapid generation of molecular complexity is achieved by coupling different catalytic metalcarbene transfer reactions (chemoselective cross-metathesis followed by tandem carbonyl ylide formation–cycloaddition) in a one-flask operation.



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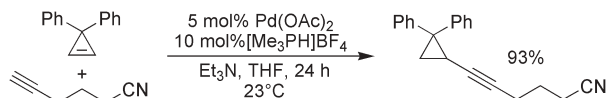


Lithocholic acid analogues, new and potent α -2,3-sialyltransferase inhibitors

Kai-Hsuan Chang, Linselot Lee, Jessica Chen and Wen-Shan Li*

A new type of noncompetitive α -2,3-sialyltransferase inhibitor has been synthesized; the authors report the discovery, preparation and inhibitory activity of sixteen lithocholic acid analogues.

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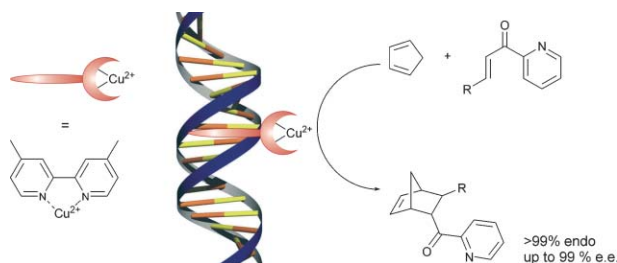


Palladium-catalyzed addition of alkynes to cyclopropenes

Jiandong Yin and John D. Chisholm*

Palladium-catalyzed addition of alkynes to cyclopropenes occurs in high yield under mild conditions without the need to protect many sensitive functional groups (aldehydes and carboxylic acids).

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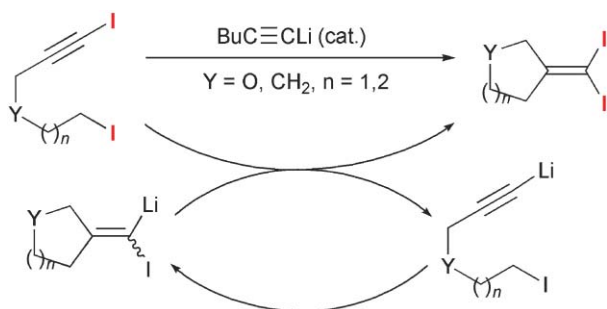


Highly enantioselective DNA-based catalysis

Gerard Roelfes,* Arnold J. Boersma and Ben L. Feringa*

Very high enantioselectivities (up to 99% ee) in the copper catalyzed Diels–Alder reaction in water could be achieved using a new generation of DNA-based catalysts.

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Novel cyclization reaction of 1, ω -diiodo-1-alkynes without the loss of iodine atoms

Toshiro Harada,* Kenta Mizunashi and Keiko Muramatsu

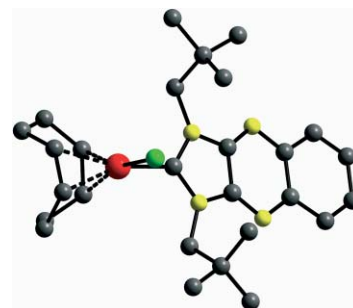
In the presence of 1-hexynyllithium, 1, ω -diiodo-1-alkynes undergo a novel cyclization reaction to give products without the loss of two iodo functional groups.

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Influence of anellation in *N*-heterocyclic carbenes: Novel quinoxaline-anellated NHCs trapped as transition metal complexes

Shanmuganathan Saravanakumar,
Markus K. Kindermann, Joachim Heinicke* and
Martin Köckerling

The synthesis and structural data of novel electron-deficient anellated imidazol-2-ylidene complexes are reported and compared with related less electron-withdrawing NHC complexes, to illustrate anellation effects.

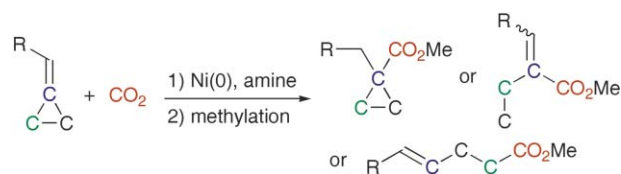


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Solvent and ligand partition reaction pathways in nickel-mediated carboxylation of methylenecyclopropanes

Masahiro Murakami,* Naoki Ishida and Tomoya Miura

Methylenecyclopropanes are carboxylated with gaseous carbon dioxide in the presence of a stoichiometric amount of a nickel complex. The reaction pathways are significantly influenced by the reaction solvent and the amine ligand.

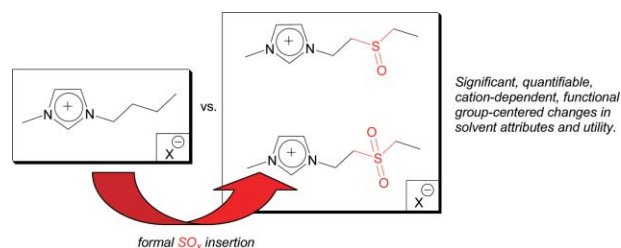


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Do ion tethered functional groups affect IL solvent properties? The case of sulfoxides and sulfones

Nawal K. Sharma, Morgan D. Tickell, Jared L. Anderson,
Joel Kaar, Veronica Pino, Benjamin F. Wicker,
Daniel W. Armstrong,* James H. Davis, Jr.* and
Alan J. Russell*

The incorporation of functional groups—specifically sulfoxide and sulfone—into imidazolium ionic liquids leads to significant, quantifiable changes in solvent parameters which have important effects on the bulk properties of the materials.

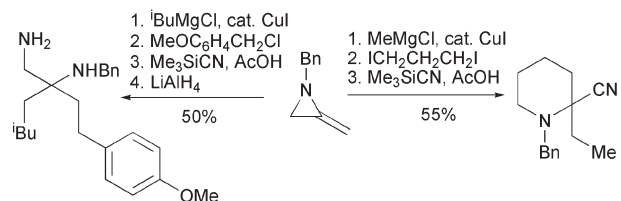


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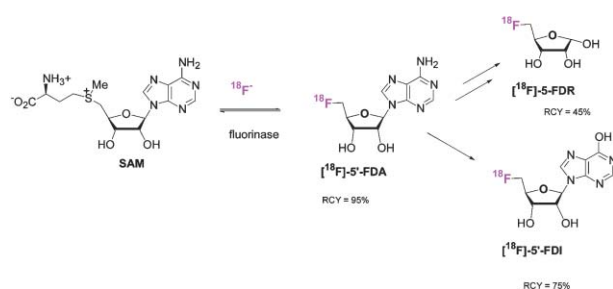
Rapid generation of molecular complexity using “hybrid” multi-component reactions (MCRs): application to the synthesis of α -amino nitriles and 1,2-diamines

Jason J. Shiers, Guy J. Clarkson, Michael Shipman* and
Jerome F. Hayes

Inspired by Strecker, new four-component reactions of methyleneaziridines have been developed that produce three intermolecular carbon–carbon bonds.



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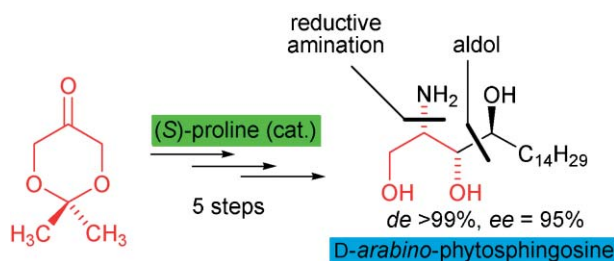


Fluorinase mediated C-¹⁸F bond formation, an enzymatic tool for PET labelling

Hai Deng, Steven L. Cobb, Antony D. Gee, Andrew Lockhart, Laurent Martarello,* Ryan P. McGlinchey, David O'Hagan* and Mayca Onega

The fluorinase enzyme from *S. cattleya* is applied as a catalyst for the efficient incorporation of [¹⁸F]-fluoride into [¹⁸F]-5'-fluoro-5'-deoxyadenosine, [¹⁸F]-5'-fluoro-5'-deoxyinosine and [¹⁸F]-5-fluoro-5-deoxyribose for positron emission tomography (PET) applications.

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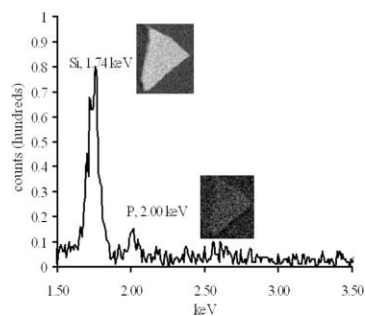


A direct organocatalytic entry to sphingoids: asymmetric synthesis of D-arabino- and L-ribo-phytosphingosine

Dieter Enders,* Jiří Paleček and Christoph Grondal

The organocatalytic asymmetric synthesis of D-arabino- and L-ribo-phytosphingosine is described employing a diastereo- and enantioselective (S)-proline-catalyzed aldol reaction of 2,2-dimethyl-1,3-dioxan-5-one and pentadecanal as the key step.

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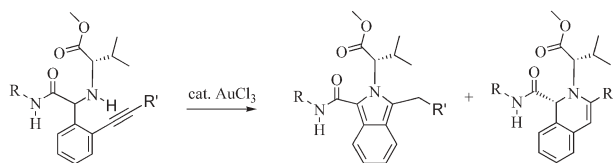


The preparation of a phosphorus doped silicon film from phosphorus containing silicon nanoparticles

Richard K. Baldwin, Jing Zou, Katherine A. Pettigrew, Gregory J. Yeagle, R. David Britt and Susan M. Kauzlarich*

Phosphorus containing silicon nanoparticles, generated by a solution reduction route under room temperature conditions for the first time, have been characterized and annealed to form a thin film.

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Isoindoles and dihydroisoquinolines by gold-catalyzed intramolecular hydroamination of alkynes

Daniel Kadzimirsz, Dirk Hildebrandt, Klaus Merz and Gerald Dyker*

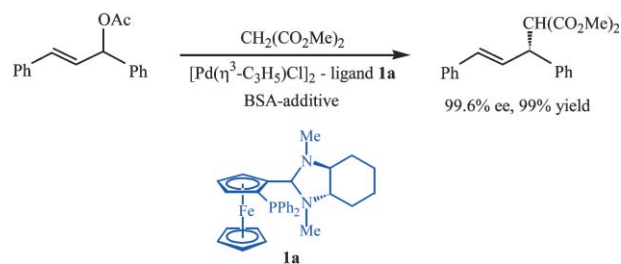
The combination of the Ugi-four-component reaction with a gold-catalyzed hydroamination gives access to chiral isoindoles and dihydroisoquinolines of high complexity in just two reaction steps.

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Highly enantioselective Pd-catalyzed allylic alkylation using new chiral ferrocenylphosphinoimidazolidine ligands

Myung-Jong Jin,* Vijay B. Takale, M. S. Sarkar and Young-Mok Kim

Ferrocenylphosphinoimidazolidine **1a** was found to act as a highly effective chiral ligand in Pd-catalyzed asymmetric allylic alkylation. Outstanding enantioselectivity as well as remarkable catalytic activity were observed.

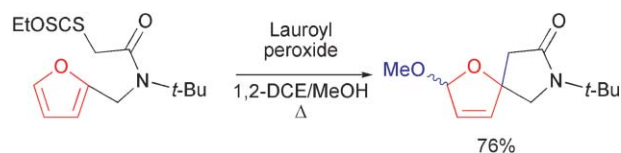


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An unusual approach to spirolactones and related structures

Soizic Guindeuil and Samir Z. Zard*

Spirocyclic structures can be obtained by an *ipso*-type radical cyclisation onto a furan or a suitably substituted pyrrole followed by oxidation of the stabilised radical adduct.

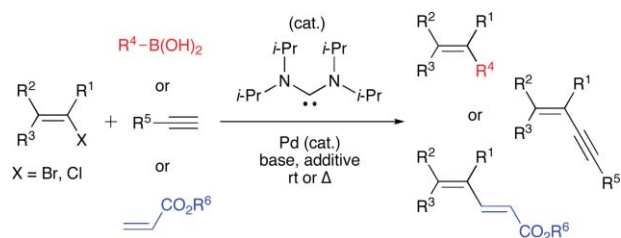


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Acyclic diaminocarbenes: simple, versatile ligands for cross-coupling reactions

Bhartesh Dhudshia and Avinash N. Thadani*

Acyclic diaminocarbenes are shown to be useful, practical ligands for several palladium-catalyzed cross-coupling reactions.

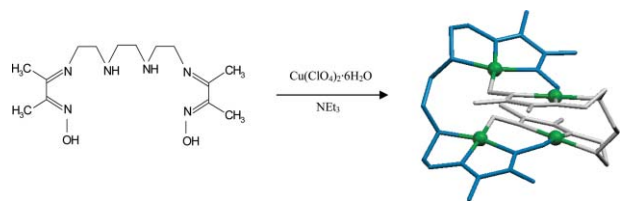


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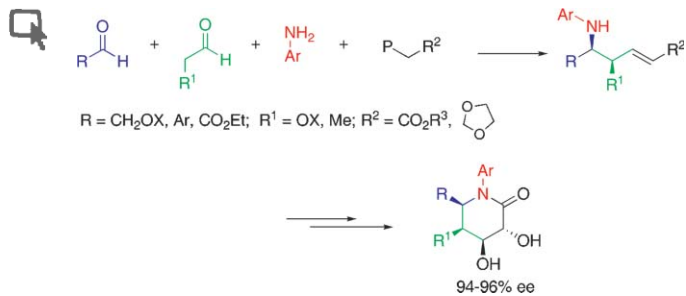
Structure and properties of a new double-stranded tetranuclear [Cu^{II}]₂ helicate

Manindranath Bera, Guillem Aromí, Wing Tak Wong and Debashis Ray*

A novel double-stranded tetranuclear helicate composed of a pair of [Cu^{II}]₂ dimers has been prepared and characterized by exploiting the flexibility, chelating ability and bridging potential of a hexadentate bis-oximate ligand.



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A concise enantioselective synthesis of iminosugar derivatives

Wei-Wei Liao, Ismail Ibrahim and Armando Córdova*

The concise *de novo* synthesis of iminosugar precursors is presented. The four stereocenters of the amino- and iminosugar derivatives are created in two-steps with high chemoselectivity and excellent enantioselectivity. This was exemplified in the short enantioselective synthesis of orthogonally protected gulo- and galactolactams that starts with an organocatalytic asymmetric Mannich–HWE or Mannich–Wittig reaction.

ADDITION AND CORRECTION

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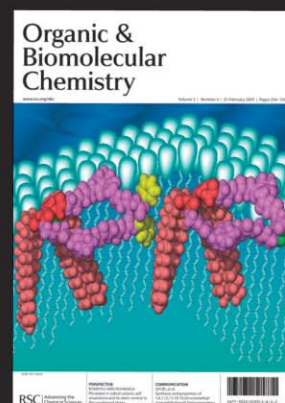
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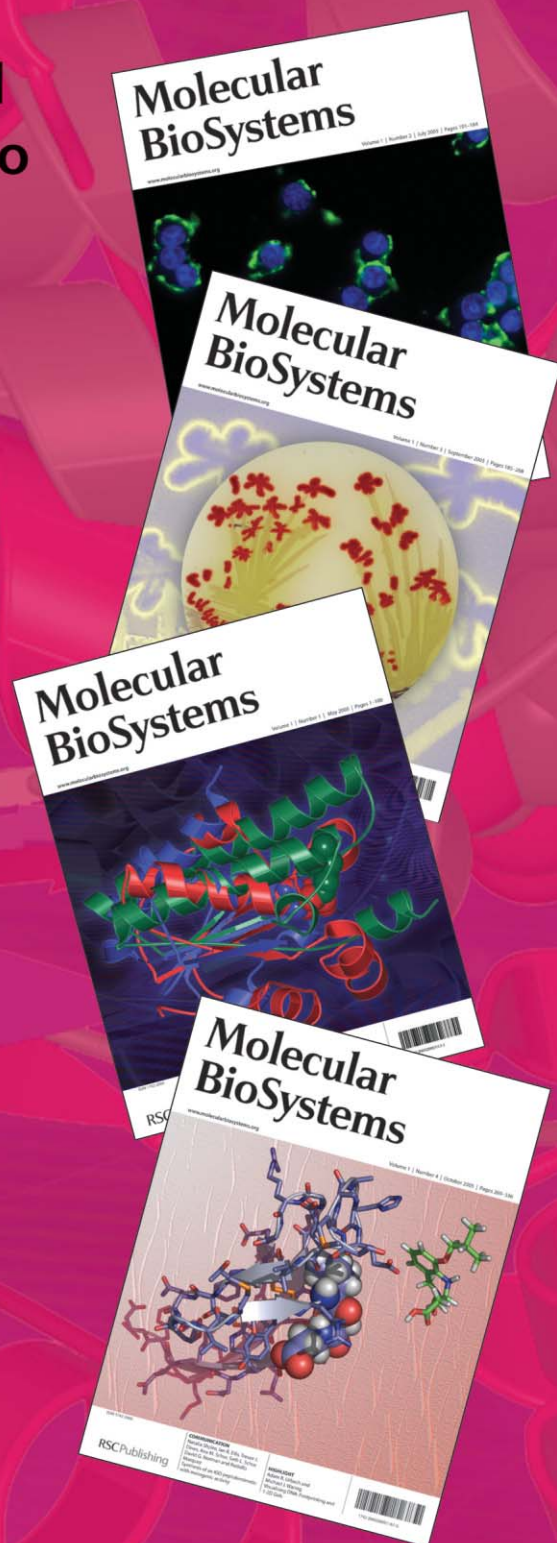
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