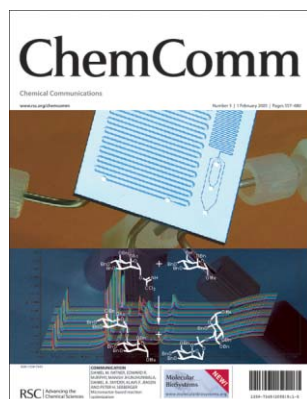


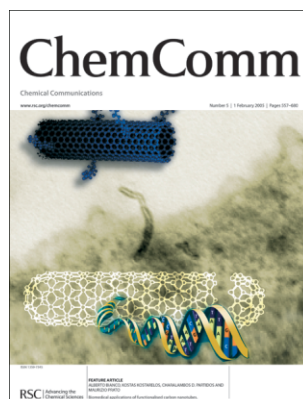
IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (5) 557-680 (2005)



Cover

Silicon and glass micro-reactor with metal tubes and connectors for efficient investigation and optimization of oligosaccharide synthesis with low use of resources and rapid throughput (centre image). Bottom portion of cover shows example chemistry superimposed on HPLC traces from of a day of experiments (~40 runs). See p. 578.



Inside cover

Functionalised carbon nanotubes penetrating through the plasma membrane of HeLa cells offer a tool for the delivery of therapeutic molecules including drugs, proteins, antigens and DNA (Image courtesy of C. Nicolaou & K. Kostarelos). See p. 571.

CHEMICAL SCIENCE

C9

Drawing together the research highlights and news from all RSC publications, *Chemical Science* provides a 'snapshot' of the latest developments across the chemical sciences showcasing newsworthy articles, as well as the most significant scientific advances.

Chemical Science

February 2005/Volume 2/Issue 2

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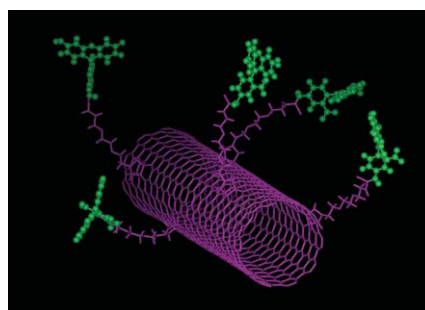
FEATURE ARTICLE

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Biomedical applications of functionalised carbon nanotubes

Alberto Bianco,* Kostas Kostarelos,*
Charalambos D. Partidos* and Maurizio Prato*

Functionalised carbon nanotubes are very interesting materials for applications in nanomedicine. This article reviews the potential of carbon nanotubes for the development of new drug, vaccine and gene delivery systems.



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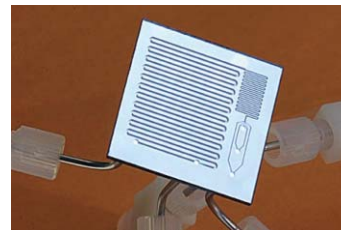
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Microreactor-based reaction optimization in organic chemistry—glycosylation as a challenge

Daniel M. Ratner, Edward R. Murphy, Manish Jhunjunwala, Daniel A. Snyder, Klavs F. Jensen* and Peter H. Seeberger*

Synthesis of complex oligosaccharides is a critical challenge to the emerging field of glycomics. This article presents a microreactor aided method for efficiently investigating and optimizing oligosaccharide synthesis with low use of resources and rapid throughput.

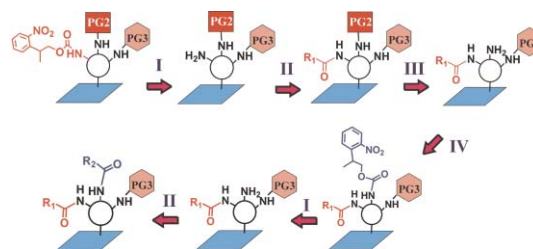


581

Photolithographic synthesis of cyclic peptide arrays using a differential deprotection strategy

Shuwei Li, Nishanth Marthandan, Dawn Bowerman, Harold R. Garner and Thomas Kodadek*

A new method to produce a cyclic peptide library that overcomes many problems encountered by current approaches.

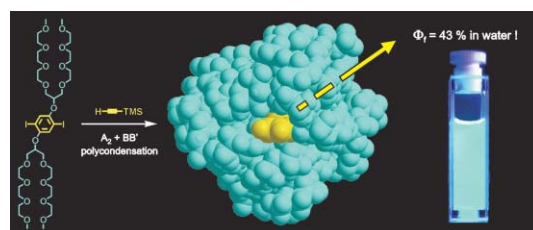


584

Practical synthesis of an amphiphilic, non-ionic poly(*para*-phenyleneethynylene) derivative with a remarkable quantum yield in water

Anzar Khan, Stephan Müller and Stefan Hecht*

A convenient $A_2 + BB'$ polycondensation route has been used to prepare poly(*para*-phenyleneethynylene)s carrying branched oligoethyleneglycol side chains that efficiently encapsulate the polymer backbone in a polar shell to provide both solubility and exceptionally high photoluminescence efficiency in aqueous environments.

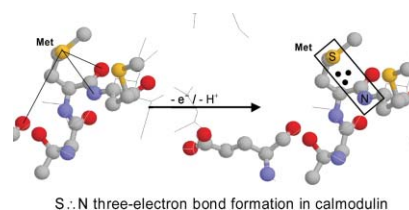


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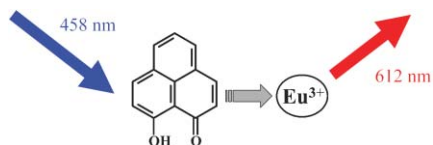
Calmodulin methionine residues are targets for one-electron oxidation by hydroxyl radicals: formation of S \cdot :N three-electron bonded radical complexes

Thomas Nauser, Michael Jacoby, Willem H. Koppenol, Thomas C. Squier and Christian Schöneich

Time-resolved optical spectroscopy detected sulfur–nitrogen three-electron bonded radicals during the one-electron oxidation of calmodulin by hydroxyl radicals. These species formed with wild-type calmodulin but not mutant calmodulin, in which several methionine residues were replaced with leucine.



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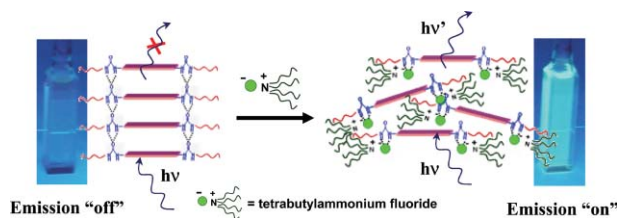


Visible light sensitisation of europium(III) luminescence in a 9-hydroxyphenal-1-one complex

Rik Van Deun,* Peter Nockemann, Pascal Fias, Kristof Van Hecke, Luc Van Meervelt and Koen Binnemans

The 9-hydroxyphenal-1-one ligand forms stable 3 : 1 complexes with trivalent lanthanides, in which it acts as an antenna suitable for the visible light excitation (up to 475 nm) of the trivalent europium ion.

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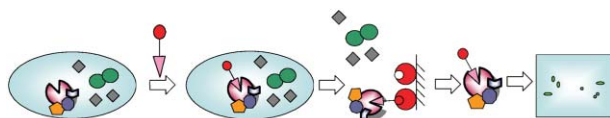


Anion induced modulation of self-assembly and optical properties in urea end-capped oligo(*p*-phenylenevinylene)s

Reji Varghese, Subi J. George and Ayyappanpillai Ajayaghosh*

H-Bond and π -stack assisted self-assembly of urea end-capped oligo(*p*-phenylenevinylene)s resulted in non-emissive one-dimensional aggregates which upon addition of F^- facilitate breakage to a strongly emissive non-aggregated state.

596

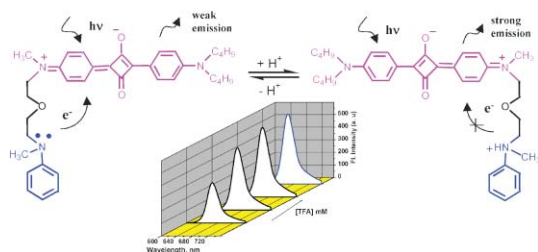


A proteomic strategy for the identification of caspase-associated proteins

Eunice L. P. Tan, Resmi C. Panicker, Grace Y. J. Chen and Shao Q. Yao*

We report the efficient *in vivo* labeling of caspases expressed inside apoptotic HeLa cells using fluoromethyl ketone (fmk)-containing probes. Preliminary results indicated that these probes may be used to identify caspase-associated proteins.

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Proton controlled intramolecular photoinduced electron transfer (PET) in podand linked squaraine-aniline dyads

Easwaran Arunkumar and Ayyappanpillai Ajayaghosh*

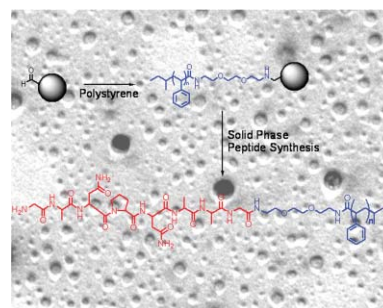
Proton controlled intramolecular PET signaling in podand linked squaraine-aniline dyads is described. This is likely the first example where a squaraine moiety behaves as a switchable electron acceptor and is useful in the design of ion probes.

602

Solid phase synthesis of biohybrid block copolymers

Irene C. Reynhout, Dennis W. P. M. Löwik,
Jan C. M. van Hest, Jeroen J. L. M. Cornelissen* and
Roeland J. M. Nolte

The solid phase synthesis of biohybrid block copolymers is described by first attaching a hydrophobic polymer to the resin and subsequently growing the desired peptide sequence using standard coupling chemistry.

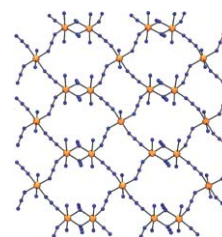


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A two-dimensional azido-based topologic ferrimagnet

Albert Escuer,* Franz A. Mautner, Mohamed A. S. Goher,
Morsy A. M. Abu-Youssef and Ramon Vicente

Ferrimagnetic behaviour with $T_c = 18$ K based on topological reasons, has been characterized in a new class of homometallic 2-D Mn^{II}/azido network.

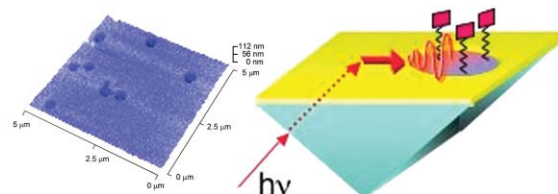


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Effective photoexcitation in gold nanowells based on localized surface plasmon

Akito Ishida* and Ayako Fujii

The localization of the surface plasmon (SP) field in nanospace using wavelength-sized gold wells prepared on a glass substrate and its application to excite fluorophores immobilized on the bottom surfaces have been studied.

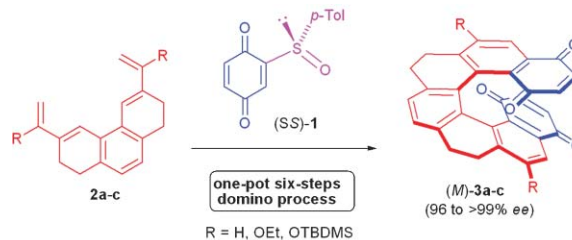
Effective Excitation in Gold Nanowells

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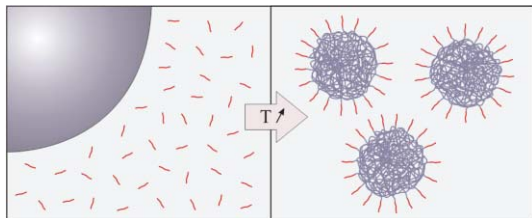
Efficient asymmetric synthesis of [7]helicene bisquinones

M. Carmen Carreño,* Marcos González-López and
Antonio Urbano*

The efficient one-pot six-step domino process which occurs when (SS)-2-(*p*-tolylsulfinyl)-1,4-benzoquinone (**1**) reacts with 3,6-divinyl-1,2,7,8-tetrahydrophenanthrenes **2a-c** allowed enantioselective access to [7]helicene bisquinones **3a-c** with excellent optical purities (96 to > 99% *ee*).



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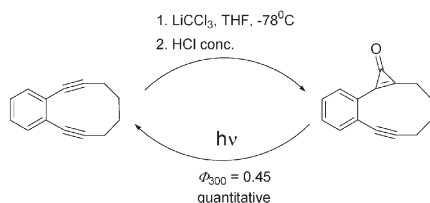


Surfactant-free synthesis of amphiphilic diblock copolymer nanoparticles *via* nitroxide-mediated emulsion polymerization

Guillaume Delaittre, Julien Nicolas, Catherine Lefay, Maud Save and Bernadette Charleux*

Nitroxide-terminated water-soluble poly(sodium acrylate) was used as a macroinitiator for the batch polymerization of styrene and *n*-butyl acrylate in water. Controlled polymerization was observed and the system led to stable, 20 wt% solids suspensions of amphiphilic diblock copolymer nanoparticles.

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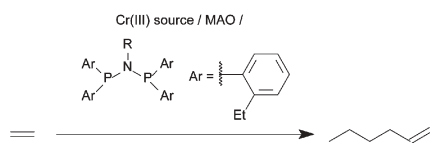


Photoswitchable enediynes: use of cyclopropanone as photocleavable masking group for the enediyne triple bond

Andrei Poloukhine and Vladimir V. Popik*

Cyclopropanone **1**, 2,3-benzobicyclo[8.1.0]undec-1(10)-en-4-yn-11-one, is a thermally stable compound showing no signs of decomposition after heating at 84 °C for 7 d. The UV irradiation of **1** results in an efficient ($\Phi_{300} = 0.45$) and quantitative formation of benzannelated enediyne **2**, which undergoes Bergman cyclization at the above temperature.

620

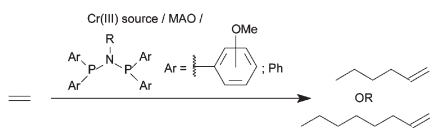


Highly selective chromium-based ethylene trimerisation catalysts with bulky diphosphinoamine ligands

Kevin Blann,* Annette Bollmann, John T. Dixon, Fiona M. Hess, Esna Killian, Hulisani Maumela, David H. Morgan, Arno Neveling, Stephanus Otto and Matthew J. Overett

In situ prepared chromium catalysts containing bulky diphosphinoamine (PNP) ligands, upon activation with MAO, are extremely efficient catalysts for the trimerisation of ethylene to 1-hexene.

622



Ethylene trimerisation and tetramerisation catalysts with polar-substituted diphosphinoamine ligands

Matthew J. Overett,* Kevin Blann, Annette Bollmann, John T. Dixon, Fiona Hess, Esna Killian, Hulisani Maumela, David H. Morgan, Arno Neveling and Stephanus Otto

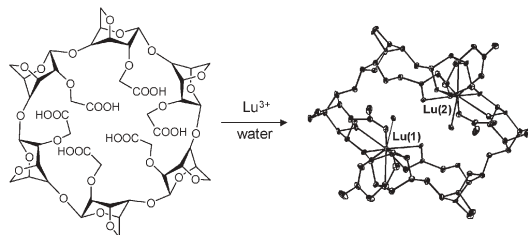
Chromium-based catalyst systems with polar-substituted diphosphinoamine ligands are selective for either trimerisation or tetramerisation of ethylene, depending on the position of the polar groups on the aryl rings.

625

Inclusion complexes of trivalent lutetium cations with an acidic derivative of per(3,6-anhydro)- α -cyclodextrin

Célia Bonnet, Andrée Gabelle, Jacques Pécaut, Pascal H. Fries and Pascale Delangle*

The crystal structure of $[\text{Lu}_2(\text{ACX})(\text{H}_2\text{O})_2]$ is the first example of a lanthanide–cyclodextrin inclusion complex; solution studies have led to a predictive speciation model allowing the control of the number of lutetium encapsulated in the cavity.

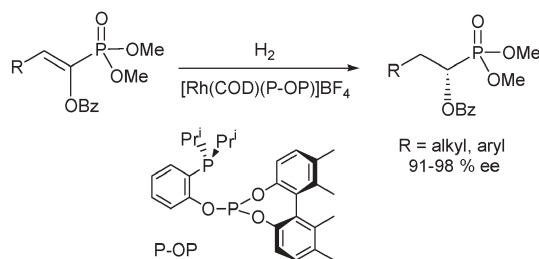


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Highly enantioselective hydrogenation of enol ester phosphonates catalyzed by rhodium phosphine-phosphite complexes

Miguel Rubio, Andrés Suárez, Eleuterio Álvarez and Antonio Pizzano*

Modularly designed chiral phosphine-phosphites have been applied in the highly enantioselective catalytic hydrogenation of several β -alkyl and β -aryl enol benzoate phosphonates.

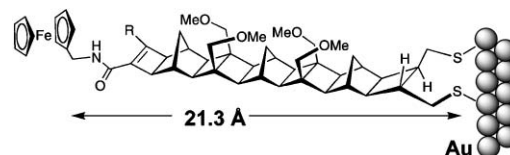


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Unusually rapid heterogeneous electron transfer through a saturated bridge 18 bonds in length

Jingquan Liu, J. Justin Gooding* and Michael N. Paddon-Row*

Rapid rates of electron transfer are observed through a stable, saturated and rigid norbornylogous bridge, 21.3 Å long. Electron transfer rates are 1000 times greater than the equivalent length alkanethiol.

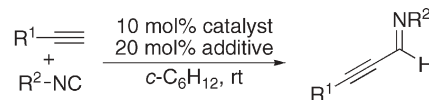


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Direct mono-insertion of isocyanides into terminal alkynes catalyzed by rare-earth silylamides

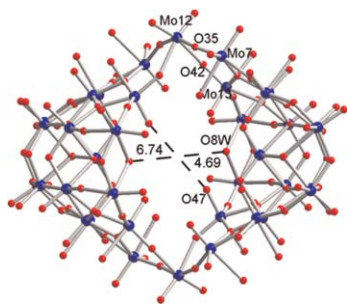
Kimihiko Komeyama,* Daisuke Sasayama, Tomonori Kawabata, Katsuomi Takehira and Ken Takaki

Rare-earth silylamides, $\text{Ln}[\text{N}(\text{SiMe}_3)_2]_3$, are shown to be effective in the catalytic direct mono-insertion of isocyanides into terminal alkynes, which has no precedent by transition metal catalysts.



catalyst = $\text{Ln}[\text{N}(\text{SiMe}_3)_2]_3$ (Ln = Y, La, Sm, Yb)

637

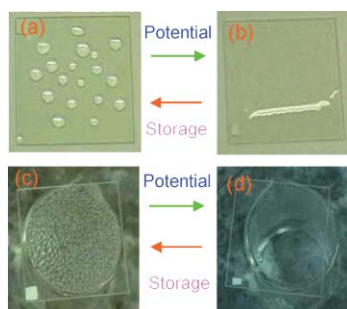


A mesoporous hydrogen-bonded organic–inorganic framework bearing the isopolymolybdate $[\text{Mo}_{36}\text{O}_{112}(\text{OH}_2)_{16}]^{8-}$

Reinaldo Atencio,* Alexander Briceño and Xacvier Galindo

The mesoporous H-bonded organic–inorganic hybrid material displays large interconnected cavities and exhibits a reversible water sorption behaviour.

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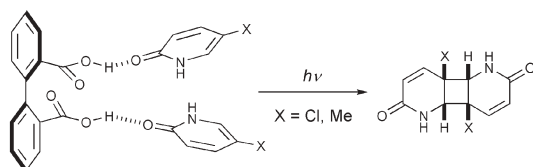


Electrochemically generated super-hydrophilic surfaces

JeromeRajan Premkumar and Soo Beng Khoo*

Purely electrochemical generation of super-hydrophilic surfaces is achieved at $\text{In}_2\text{O}_3\text{-SnO}_2$ surfaces by application of high anodic potentials for a prolonged period in the absence of ultraviolet light irradiation.

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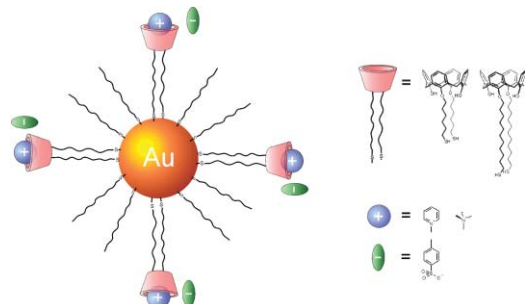


A new [2 + 2] photodimerization of 5-chloro- and 5-methyl-2-pyridone in their inclusion complexes with 1,1'-biphenyl-2,2'-dicarboxylic acid as a model for DNA damage by photodimerization of its thymine component

Shinya Hirano, Shinji Toyota and Fumio Toda*

As a model for DNA damage by photodimerization of its thymine component, a new [2 + 2] photodimerization of 5-chloro and 5-methyl-2-pyridone to the corresponding *cis-anti*-dimers as their inclusion complexes with 1,1'-biphenyl-2,2'-dicarboxylic acid was found, and the mechanism of this stereoselective solid state reaction was studied by X-ray analysis.

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Recognition of quaternary ammonium cations by calix[4]arene derivatives supported on gold nanoparticles

Arturo Arduini, Domenico Demuru, Andrea Pochini* and Andrea Secchi

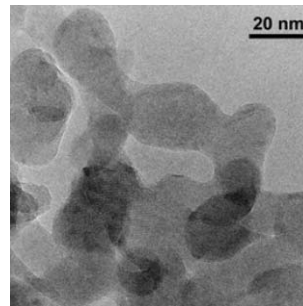
Calix[4]arene derivatives supported on monolayer protected gold clusters show a remarkable enhancement of binding efficiency in the recognition of quaternary ammonium salts in apolar media.

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Flame synthesis of calcium carbonate nanoparticles

Matthias Huber, Wendelin J. Stark,* Stefan Loher, Marek Maciejewski, Frank Krumeich and Alfons Baiker

Calcium carbonate nanoparticles of 20–50 nm size were obtained from a flame spray process where combustion of specific calcium-containing precursors results in amorphous or crystalline calcium carbonate particles depending on the spray flow conditions.

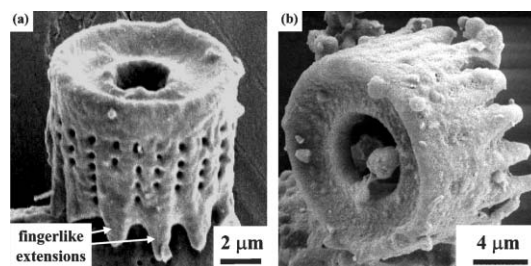


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Sol-gel synthesis on self-replicating single-cell scaffolds: applying complex chemistries to nature's 3-D nanostructured templates

Michael R. Weatherspoon, Shawn M. Allan, Eden Hunt, Ye Cai and Kenneth H. Sandhage*

A sol-gel process was used, for the first time, to apply a multi-component, nanocrystalline, functional ceramic compound (BaTiO_3 , shown in (b)) to a three-dimensional, self-replicating scaffold (shown in (a)) derived from a single-celled micro-organism (a diatom).

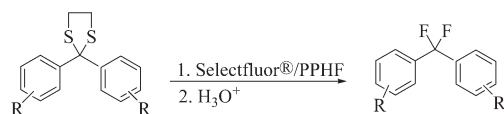


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***gem*-Difluorination of 2,2-diaryl-1,3-dithiolanes by Selectfluor® and pyridinium polyhydrogen fluoride**

V. Prakash Reddy,* Ramesh Alleti, Meher K. Perambuduru, Urs Welz-Biermann, Herwig Buchholz and G. K. Surya Prakash*

2,2-Diaryl-1,3-dithiolanes, readily available from diaryl ketones, were transformed into the corresponding *gem*-difluoromethylene compounds using the reagent combination, Selectfluor® and pyridinium polyhydrogen fluoride (PPHF), under mild conditions in moderate to good yields.



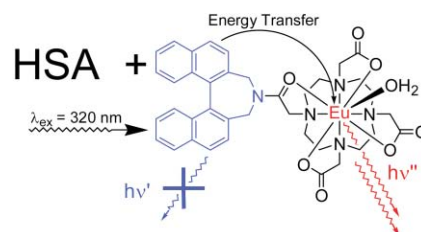
R = c.g., H, F, Cl, Br, OMe

657

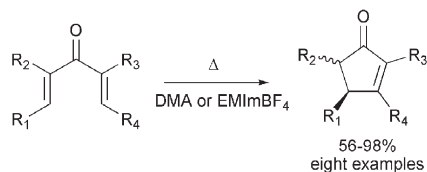
A binaphthyl-containing Eu(III) complex and its interaction with human serum albumin: a luminescence study

Jacqueline Hamblin, Neil Abboyi and Mark P. Lowe*

On binding to human serum albumin (HSA) the Eu(III) luminescent emission enhancement of a complex containing a binaphthyl chromophore enables the determination of binding constants.



660

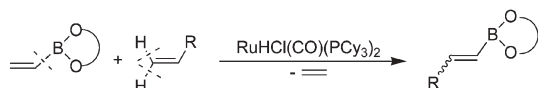


Reagent-free Nazarov cyclisations

Frederic Douelle, Lauren Tal and Michael F. Greaney*

Substituted dienones undergo Nazarov cyclisation when heated in DMA or an ionic liquid. No Lewis acids are necessary, and the resulting cyclopentenones are produced cleanly in good yields.

663

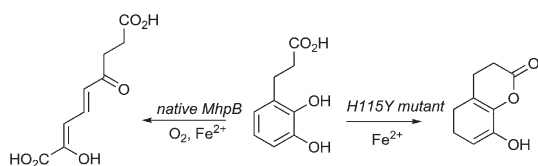


New catalytic route to functionalized vinylboronates

Bogdan Marciniak,* Magdalena Jankowska and Cezary Pietraszuk

Vinylsubstituted boronates *i.e.* vinyldioxaborolane and vinyldioxaborinane react regioselectively with olefins in the presence of $\text{RuHCl}(\text{CO})(\text{PCy}_3)_2$ with the formation of functionalized vinylboron derivatives.

666

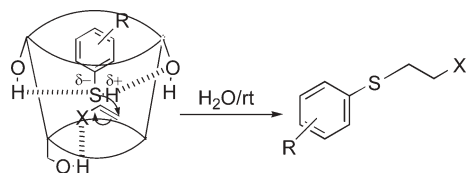


Lactone synthesis activity in a site-directed mutant of an extradiol catechol dioxygenase enzyme

Sharon Mendel, Andrew Arndt and Timothy D. H. Bugg*

A H115Y site-directed mutant of extradiol catechol dioxygenase MhpB catalyses an intramolecular lactonisation reaction upon its natural substrate.

669



Study of the Michael addition of β -cyclodextrin–thiol complexes to conjugated alkenes in water

N. Srilakshmi Krishnaveni, K. Surendra and K. Rama Rao*

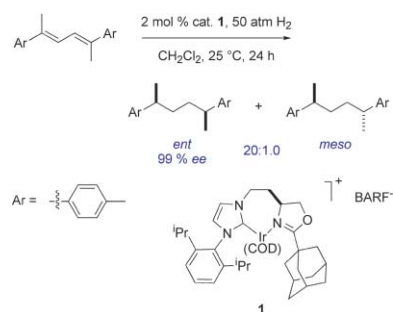
Supramolecular catalysis of the Michael addition of thiols to α,β -unsaturated compounds proceeds in the β -cyclodextrin cavity with the thiol nucleophile at the secondary side approaching the elegantly set olefin at the primary side.

672


Stereoselective hydrogenations of aryl-substituted dienes

Xiuhua Cui, James W. Ogle and Kevin Burgess*

Iridium carbene oxazoline complex **1** can mediate asymmetric hydrogenations of dienes to give two chiral centers. In the most favorable cases, high conversions, yields, *ent/meso* diastereoselectivities, and enantioselectivities were obtained.

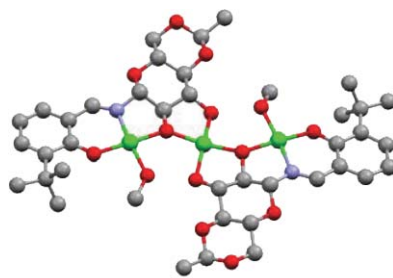


675

 **Trinuclear coordinatively labile Cu(II) complex of 4,6-*O*-ethylidene-β-D-glucopyranosylamine derived Schiff base ligand and its reactivity towards primary alcohols and amines**

Ajay K. Sah, Merii Kato and Tomoaki Tanase*

A glucose derived novel coordinatively labile, alcoholophilic linear trinuclear Cu(II) complex has been synthesised and structurally characterised, which activates the C–Cl bond in the presence of methylamine at room temperature



Cu (green), O (red), N (blue), C (gray)


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