



Cover
The microencapsulated catalyst PEM-MC
OsO₄.



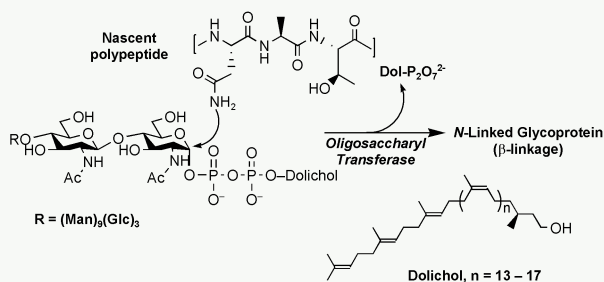
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www.rsc.org/chembiol

contents

FOCUS ARTICLE

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A chemist's approach to biochemical complexity



Professor Barbara Imperiali of MIT discusses her work at the interface between chemistry and biology, studying the structure and function of complex biological molecules, pathways and systems.

FEATURE ARTICLE

449

Renaissance of immobilized catalysts. New types of polymer-supported catalysts, 'microencapsulated catalysts', which enable environmentally benign and powerful high-throughput organic synthesis

Shū Kobayashi* and Ryo Akiyama



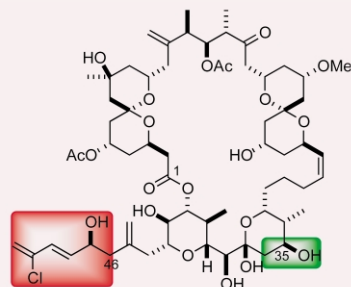
Microencapsulated Sc, Os, Pd, and Ru catalysts were prepared and high activities attained. No leaching of the catalysts occurred, and the immobilized catalysts were recovered quantitatively by simple filtration and reused without loss of activity. This method enables direct immobilization of metals onto polymers, and stabilization of normally unstable species such as Pd(0)(PPh₃).

COMMUNICATIONS

462

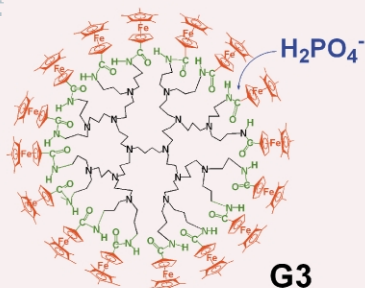
Synthesis and biological evaluation of spongistatin/altohyrtin analogues: E-ring dehydration and C46 side-chain truncation

Ian Paterson,* Jose L. Aceña, Jordi Bach, David Y.-K. Chen and Mark J. Coster



Two fully synthetic analogues of the potent antimetabolic marine macrolide spongistatin 1/altohyrtin A were prepared and evaluated as growth inhibitory agents against a range of human tumour cell lines.

464



Redox-robust pentamethylamidoferrocenyl metallodendrimers that cleanly and selectively recognize the H_2PO_4^- anion

Jaime Ruiz, Maria Jesus Ruiz Medel, Marie-Christine Daniel, Jean-Claude Blais and Didier Astruc*

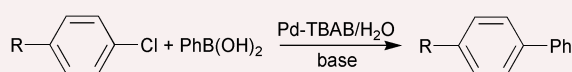
Pentamethylamidoferrocenyl dendrimers (five generations) have lipophilic pentamethylcyclopentadienyl groups and are stable under both the Fe^{II} and Fe^{III} forms; both properties greatly facilitate their use as selective H_2PO_4^- supramolecular electrochemical sensors.

466

The Suzuki coupling of aryl chlorides in TBAB–water mixtures

Robin B. Bedford,* Michael E. Blake, Craig P. Butts* and Debbie Holder

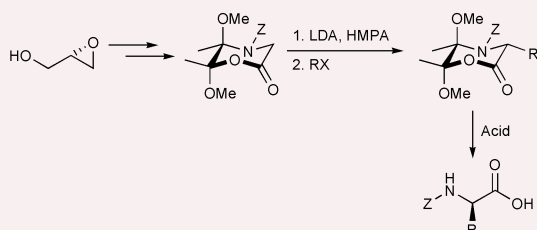
Simple ‘ligand free’ catalysts derived from palladium acetate in mixtures of TBAB and water are effective in the Suzuki coupling of deactivated aryl chloride substrates.



468

A 2,3-butanedione protected chiral glycine equivalent—a new building block for the stereoselective synthesis of enantiopure *N*-protected α -amino acids

Darren J. Dixon, Christopher I. Harding, Steven V. Ley* and D. Matthew G. Tilbrook

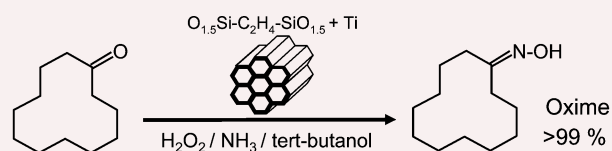


A new chiral glycine equivalent has been synthesised from glycidol using a chiral memory protocol, and its use in the synthesis of *N*-*Z* protected α -amino acids was demonstrated in a series of diastereoselective lithium enolate alkylation reactions and subsequent acid hydrolyses.

470

Amoximation of ketones catalyzed by titanium-containing ethane bridged hybrid mesoporous silsesquioxane

Asim Bhaumik, Mahendra P. Kapoor and Shinji Inagaki*



The first example of amoximation of bulky ketones over highly hydrophobic titanium incorporated ethane bridged hybrid mesoporous silsesquioxane with high selectivity of oximes is presented.

472

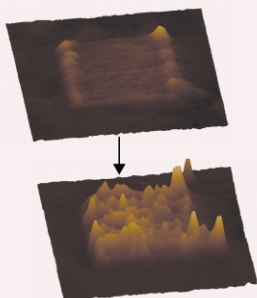


Molecular tectonics: infinite cationic double stranded helical coordination networks

Abdelaziz Jouaiti, Mir Wais Hosseini* and Nathalie Kyritsakas

Self-assembly of tectons bearing two pyridines interconnected by a polyethyleneglycol spacer and silver cation leads to the formation of infinite double helices in the crystalline phase.

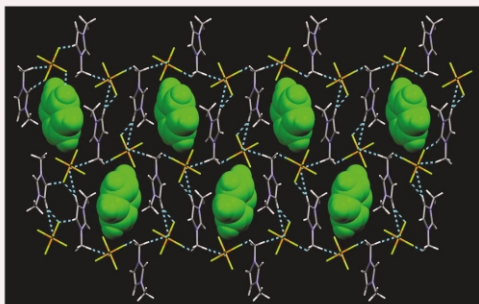
474

**Systematic manipulation of surface chemical reaction on the nanoscale: a novel approach for constructing three-dimensional nanostructures**

Xiuzhu Wang, Dejian Zhou, Trevor Rayment and Chris Abell*

Nanoscale patches, created by nanografting a maleimide-terminated thiol into a self-assembled monolayer, were elaborated by sequential chemical reactions. Each stage in the nanofabrication was followed by atomic force microscopy, providing a controlled approach to the fabrication of novel three-dimensional surface nanostructures.

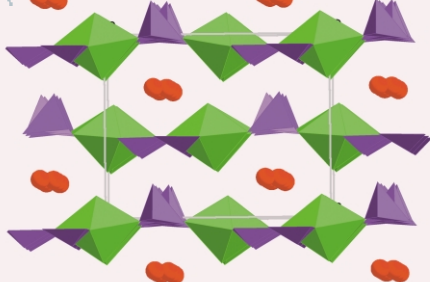
476

**Liquid clathrate formation in ionic liquid–aromatic mixtures**

John D. Holbrey, W. Matthew Reichert, Mark Nieuwenhuyzen, Oonagh Sheppard, Christopher Hardacre and Robin D. Rogers*

Liquid clathrate formation appears as a general phenomena when common ionic liquids are mixed with aromatic hydrocarbons. In the system 1,3-dimethylimidazolium hexafluorophosphate–benzene, the aromatic solute was also trapped as a crystalline 2:1 inclusion compound.

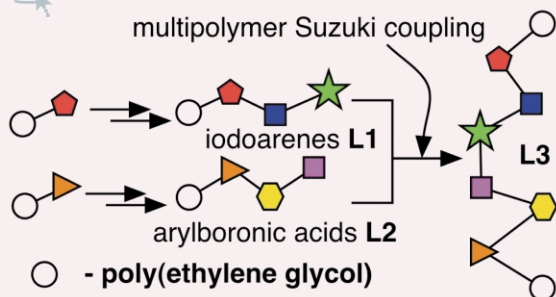
478

**Structural characterization of the first hydrothermally synthesized plutonium compound, PuO₂(IO₃)₂·H₂O**

Wolfgang Runde,* Amanda C. Bean, Thomas E. Albrecht-Schmitt and Brian L. Scott

The synthesis and characterization of a new plutonium iodate, PuO₂(IO₃)₂·H₂O, represents the first single crystal structure of a plutonyl(vi) compound. The layered structure is built from corner-sharing [IO₃] and [PuO₇] units and differs structurally from the hydrated uranyl(vi) iodate, UO₂(IO₃)₂(H₂O).

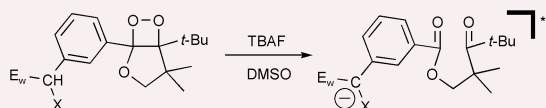
480

**Soluble polymer-supported convergent parallel library synthesis**

Jung-Mo Ahn, Paul Wentworth Jr.* and Kim D. Janda*

Soluble polymer-supported convergent synthesis has for the first time been successfully exploited for parallel library synthesis. Sub-libraries of tripeptide iodoarenes **L1** and arylboronic acids **L2** reacted smoothly in a multipolymer Pd^{II}-catalyzed Suzuki coupling reaction to generate a library of bisaryl-linked hexapeptides **L3**.

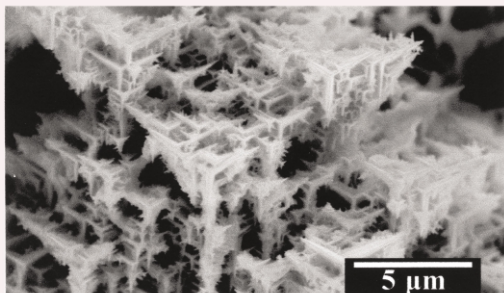
482

**Fluoride-induced chemiluminescent decomposition of 1,2-dioxetanes bearing a phenyl moiety substituted with a methyl having an electron-withdrawing group**

Masakatsu Matsumoto,* Toshiyuki Mizuno and Nobuko Watanabe

A new type of dioxetane bearing a phenyl moiety substituted with a methyl having an electron-withdrawing group decomposed through an unstable dioxetane bearing a benzylic carbanion to afford crimson to yellow light on treatment with TBAF.

484

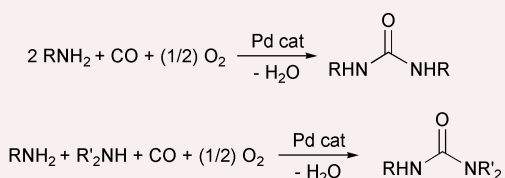


Self-organized formation of a hierarchical self-similar structure with calcium carbonate

Hiroaki Imai,* Tomohiro Terada and Satoshi Yamabi

A hierarchical self-similar morphology over wide length scales ranging from nano- to macro-scales was created through self-organized growth in a supersaturated solution with a gradual increase in the influence of silicate anions.

486

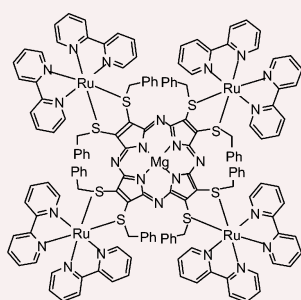


A novel and efficient method for the Pd-catalysed oxidative carbonylation of amines to symmetrically and unsymmetrically substituted ureas

Bartolo Gabriele,* Raffaella Mancuso, Giuseppe Salerno* and Mirco Costa

A very efficient method for the synthesis of symmetrically and unsymmetrically substituted ureas by direct Pd-catalyzed oxidative carbonylation of amines has been developed.

488

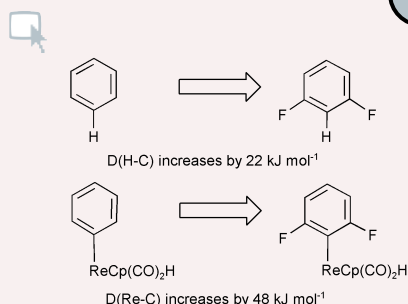


Synthesis, spectral and electrochemical investigations of bichromophoric pentads possessing tetraazaporphyrin and (bipy)₂Ru^{II}/(phen)₂Ru^{II} moieties

Rajendra Prasad,* Elisa Murguly and Neil R. Branda

Tethering four (bpy)₂Ru^{II} or (phen)₂Ru^{II} moieties to [Mg(TAP)] core through thioether coordination leads to introduction of a low lying oxidation state, a shift in the MLCT absorption band and the channellization of the (bpy)₂Ru^{II} MLCT and LMCT absorption energies to the [Mg(TAP)] centered S₂ emission.

490

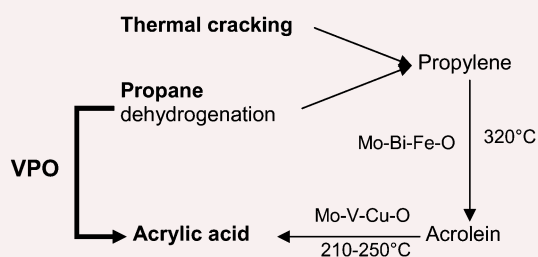


Bond energy M–C/H–C correlations: dual theoretical and experimental approach to the sensitivity of M–C bond strength to substituents

Eric Clot, Maria Besora, Feliu Maseras, Claire Mégret, Odile Eisenstein,* Beatriz Oelckers and Robin N. Perutz*

Metal–carbon bond energies, $D(\text{M}-\text{C})$, in fluoroaryl derivatives of rhenium are calculated to increase in energy more than twice as fast as the corresponding H–C bond energies; DFT calculations form an efficient tool to establish the correlations between bond energies.

492

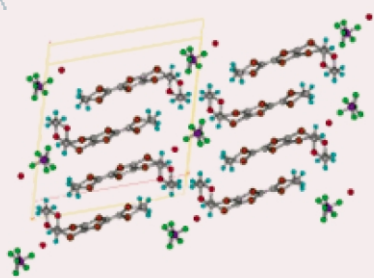


Effect of water on the catalytic behaviour of VPO in the selective oxidation of propane to acrylic acid

G. Landi,* L. Lisi and J. C. Volta

Evaluation of structural modifications of vanadyl pyrophosphate under different gas mixtures and determination of the best concentration of water vapour in the feed for maximising acrylic acid yield in the mild oxidation of propane were effected.

494



A new organic superconductor, (DODHT)₂BF₄·H₂O

Hiroyuki Nishikawa,* Asami Machida, Takanobu Morimoto, Koichi Kikuchi,* Takeshi Kodama, Isao Ikemoto, Jun-ichi Yamada,* Harukazu Yoshino and Keizo Murata

A new organic superconductor derived from DODHT [(1,4-dioxane-2,3-diylthio)dihydratetrafulvalene], the tetrahedral anion BF₄⁻ and one water molecule has been prepared, and exhibits a superconducting transition temperature of 3.2 K under a hydrostatic pressure of 15.5 kbar.

496

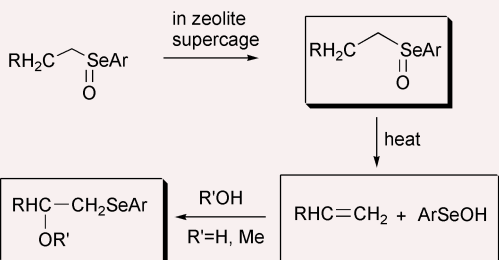


A novel approach to polymeric hollow nanospheres with stabilized structure

Min Kuang, Hongwei Duan, Jing Wang, Daoyong Chen and Ming Jiang*

Polymeric hollow nanospheres with stabilized structure were obtained by self-assembly of rod-like PAE and coil-like poly(vinylpyridine) in THF followed by a simple photo crosslinking reaction.

498

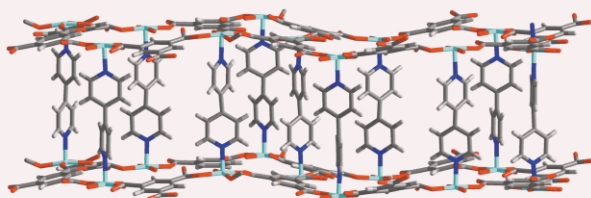


A novel role of zeolite NaY in the thermal reaction of alkyl aryl selenoxides in its supercages

Wanxuan Zhang, Haitao Yu, Yu Gao, Jiben Meng* and Teruo Matsuura

Thermal reaction of alkyl aryl selenoxides in the presence of water or methanol in the supercage of zeolite NaY was studied, and a novel role of zeolite NaY was described.

500

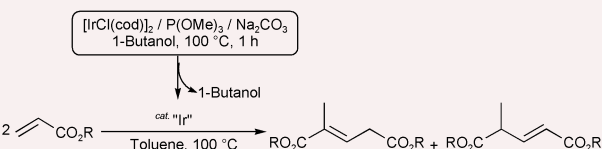


Designed layer assembly: a three-dimensional framework with 74% extra-framework volume by connection of infinite two-dimensional sheets

T. J. Prior, D. Bradshaw, S. J. Teat and M. J. Rosseinsky*

The designed connection of two-dimensional hexagonal sheets with the 4,4'-bipyridyl ligand produces a solid with large cavities and channels.

502



Head-to-tail dimerization of acrylates catalyzed by iridium complexes

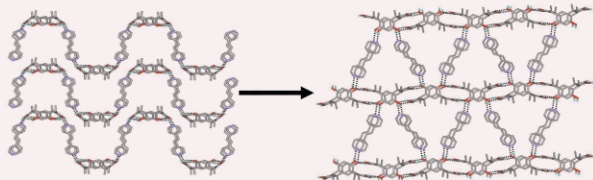
Hideto Nakagawa, Satoshi Sakaguchi and Yasutaka Ishii*

Head-to-tail dimerizations of acrylates and vinyl ketone were successfully performed by the use of iridium complexes generated *in situ* from [IrCl(cod)]₂ and alcohols in the presence of Na₂CO₃ and (MeO)₃P.

504

Transformation of a *C*-methylcalix[4]resorcinarene-based host–guest complex from a wave-like to a novel triangular brick-wall architecture

Bao-Qing Ma and Philip Coppens*

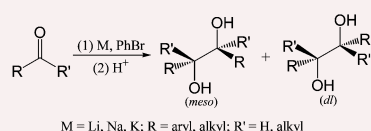


Self-assembly of *C*-methylcalix[4]resorcinarene (CMCR) and *trans*-1,4-bis(pyridyl)ethylene (bpe) in the presence of ruthenocene generates a kinetically stabilized 1D wave-like polymer which converts to a thermodynamically stabilized 2D triangular brick-wall framework upon prolonged exposure to the solution form which the crystals are obtained.

506

A novel, solventless reductive coupling of carbonyl compounds by alkali metals, catalysed by bromobenzene

Hui Zhao, De-Jin Li, Lan Deng, Lei Liu* and Qing-Xiang Guo*

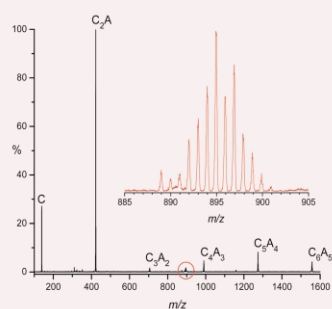


Catalysed by bromobenzene, alkali metals including Li, Na, and K can mediate the reductive coupling of carbonyl compounds under mild, solventless conditions to furnish the corresponding pinacol products.

508

Direct analysis of catalysts immobilised in ionic liquids using electrospray ionisation ion trap mass spectrometry

Paul J. Dyson,* J. Scott McIndoe* and Dongbin Zhao

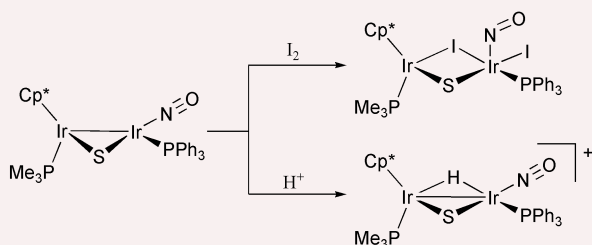


Electrospray ionisation mass spectrometry has been used to analyse transition metal catalysts that operate in ionic liquids.

510

Mono(sulfido)-bridged mixed-valence nitrosyl complex: protonation and oxidative addition of iodine across the Ir(II)–Ir(0) bond

Takanori Hattori, Shoji Matsukawa, Shigeki Kuwata,* Youichi Ishii* and Masanobu Hidaï*

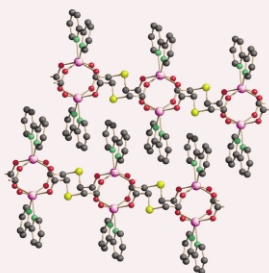


The mono(sulfido)-bridged Ir^{II}Ir⁰ nitrosyl complex [Cp*Ir(PMe₃)(μ-S)-Ir(NO)(PPh₃)] reacted with I₂ and triflic acid to give the Ir^{III}Ir^{III} diiodo complex and the Ir^{III}Ir^I hydrido complex, respectively.

512

First example of a tetra-carboxylate bridged dimanganese species

Abdessamad Grirrane, Antonio Pastor, Agustín Galindo,* Andrea Ienco, Carlo Mealli* and Patrick Rosa

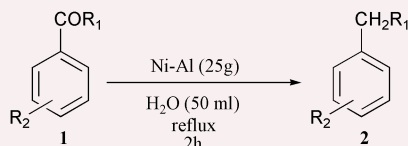


The compound [Mn(tda)(bipy)] (tda = S(CH₂COO)₂²⁻) features the first structurally characterized tetra-carboxylate dimanganese system, one of many transition metal species having the 'copper(II) acetate' core, and previously missing for manganese coordination.

514

Reduction of carbonyl groups to the corresponding methylenes with Ni–Al alloy in water

Keiko Ishimoto, Yoshiharu Mitoma, Satoko Nagashima, Hideki Tashiro, G. K. Surya Prakash, George A. Olah and Masashi Tashiro*

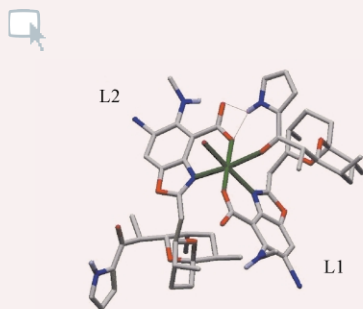


The reduction of carbonyl compounds **1a–h** using Ni–Al alloy in water under reflux proceeded to give the corresponding methylene compounds **2a–h** within 2 h in 89.0–99.8% relative yields.

516

Unusual structure of the dimeric 4-bromocalcimycin–Zn²⁺ complex

Stéphane Vila, Isabelle Canet, Jacques Guyot, Georges Jeminet* and Loïc Toupet

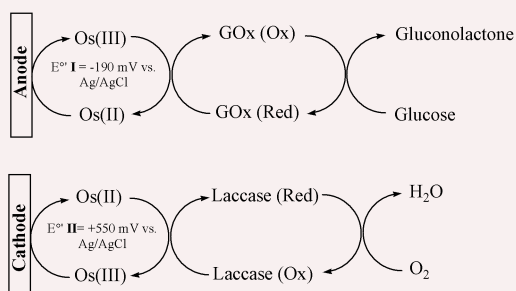


The X-ray structure of [Zn(4-bromocalcimycin)₂·H₂O] complex shows two highly different conformations (L1, L2) of the ligand in the dimeric association, unusual in this ionophore family.

518

A miniature biofuel cell operating at 0.78 V

Nicolas Mano,* Fei Mao, Woonsup Shin, Ting Chen and Adam Heller*

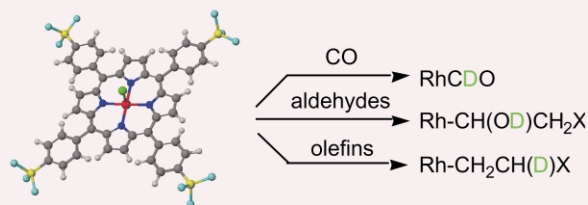


We report the highest voltage miniature biofuel cell to date, a membrane-less cell operating at 37 °C in pH 5 buffer at +0.78 V.

520

Aqueous organometallic reactions of rhodium porphyrins: equilibrium thermodynamics

Xuefeng Fu, Leah Basicckes and Bradford B. Wayland*

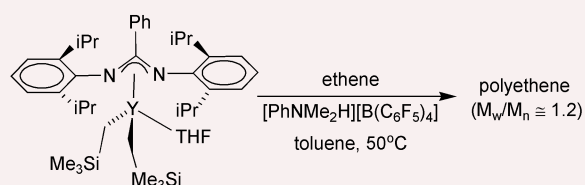


Reactivity and equilibrium thermodynamics of tetra-*p*-sulfonatophenyl porphyrin rhodium hydride in D₂O with CO, aldehydes and olefins that produce formyl, α-hydroxyalkyl and alkyl complexes have been evaluated.

522

Yttrium alkyl complexes with a sterically demanding benzamidinate ligand: synthesis, structure and catalytic ethene polymerisation

Sergio Bambirra, Daan van Leusen, Auke Meetsma, Bart Hessen* and Jan H. Teuben

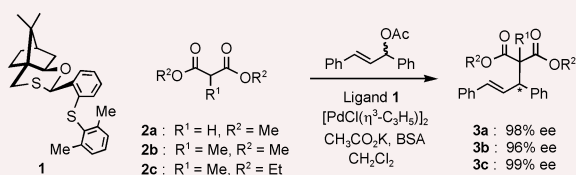


The benzamidinate yttrium dialkyl complex [PhC(NAr)₂]Y(CH₂SiMe₃)₂ (THF) (Ar = 2,6-diisopropylphenyl) reacts with [PhNMe₂H][B(C₆F₅)₄] to generate a cationic monoalkyl species that polymerises ethene to polyethene with a very narrow polydispersity.

524

Novel and efficient chiral sulfideoxathiane ligands for palladium-catalyzed asymmetric allylic alkylation

Yuko Okuyama, Hiroto Nakano,* Kouichi Takahashi, Hiroshi Hongo and Chizuko Kabuto*

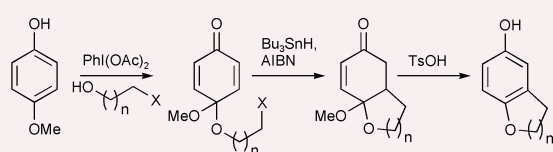


Chiral sulfideoxathiane ligand **1** showed excellent enantioselectivity up to 99% ee in the Pd-catalyzed allylic alkylation of 1,3-diphenyl-2-propenyl acetate with malonates.

526

Formal radical cyclization onto benzene rings—a general method proceeding *via* cross-conjugated dienones

Derrick L. J. Clive,* Stephen P. Fletcher and (in part) Mingzhao Zhu

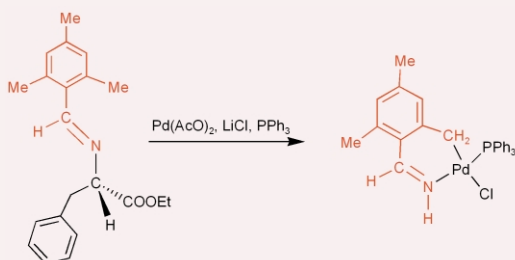


Cross-conjugated dienones bearing a haloalkoxy group, which are easily prepared from *p*-methoxyphenols, undergo radical cyclization and aromatization; the process represents formal radical closure onto a benzene ring.

528

The first NH aldimine organometallic compound. Isolation and crystal structure

Joan Albert, J. Magali Cadena, Asensio González, Jaume Granell,* Xavier Solans and Mercè Font-Bardia

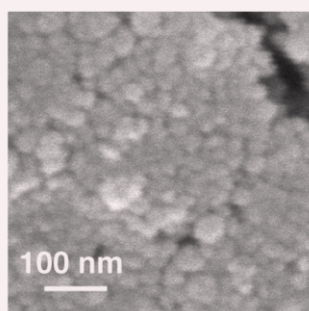


The first NH aldimine organometallic derivative is unexpectedly formed by the cleavage of the nitrogen–carbon bond of the amino acid fragment of the Schiff base 2,4,6-Me₃C₆H₂CH=NCH(CH₂Ph)COOEt when the imine is treated with palladium acetate.

530

Beta zeolite supported on silicon carbide for Friedel–Crafts fixed-bed reactions

Gauthier Winé, Joseph Matta, Jean-Philippe Tessonnier, Cuong Pham-Huu* and Marc-Jacques Ledoux

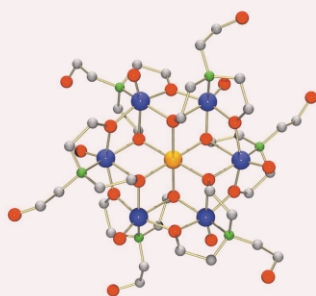


Beta zeolite supported on silicon carbide, with high thermal conductivity and high mechanical strength, was successfully used as an active and stable catalyst for Friedel–Crafts reactions in a fixed bed configuration.

532

A novel cationic heteropolyoxovanadium(IV) cluster functionalized with organic ligands: synthesis and characterization of the fully reduced species [Mn^{II}V^{IV}₆O₆{(OCH₂CH₂)₂N(CH₂CH₂OH)}₆]Cl₂

M. Ishaque Khan,* Saadia Tabussum and Robert J. Doedens*

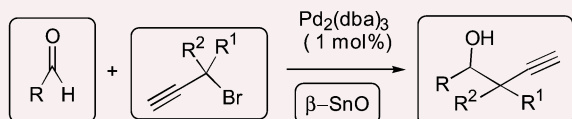


A novel cationic heteropolyoxovanadium(IV) cluster functionalized with organic ligands: synthesis and characterization of the fully reduced species [Mn^{II}V^{IV}₆O₆{(OCH₂CH₂)₂N(CH₂CH₂OH)}₆]Cl₂.

534

Palladium(0) catalyzed regioselective carbonyl propargylation across tetragonal tin(II) oxide *via* redox transmetalation

Moloy Banerjee and Sujit Roy*

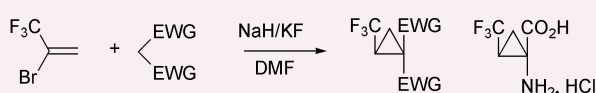


Facile synthesis of homopropargyl alcohols is achieved from propargyl bromides and aldehydes over tetragonal tin(II) oxide and catalytic palladium(0). From *in-situ* NMR and *ex-situ* IR-studies, a mechanism is proposed which involves the prior formation of dinuclear allenylpalladium followed by redox transmetalation to β -SnO.

536

A convenient stereoselective synthesis of trifluoromethyl-substituted polyfunctionalized cyclopropane: synthesis of (\pm)-*trans*-trifluoronorcoronamic acid

Biao Jiang,* Fangjiang Zhang and Wennan Xiong

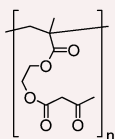


Trifluoromethylated polyfunctionalized cyclopropanes were obtained in high stereoselectivity by reacting 2-bromo-3,3,3-trifluoropropene (BrTFP) with active methylenes. This novel method was further applied to the synthesis of (\pm)-*trans* trifluoronorcoronamic acid.

538

Controlled synthesis of homopolymers and block copolymers based on 2-(acetoacetoxy)ethyl methacrylate *via* RAFT radical polymerisation

Theodora Krasia, Rémi Soula, Hans G. Börner and Helmut Schlaad*

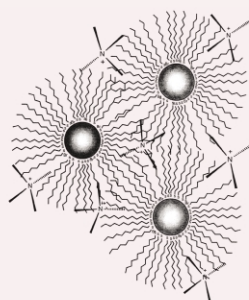


Novel types of metal ion-binding block copolymers based on 2-(acetoacetoxy)ethyl methacrylate have been synthesised by controlled RAFT radical polymerisation. These polymers have a high application potential in for example nanocasting or biomineralisation processes.

540

Purification of dodecanethiol derivatised gold nanoparticles

Cecilia A. Waters, Allan J. Mills, Kathleen A. Johnson and David J. Schiffrin*

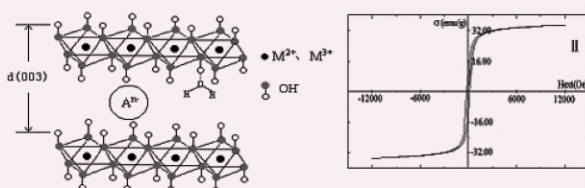


Alkane thiol capped gold nanoparticles are electrostatically stabilised in the solid phase by the adsorption of bromide and quaternary ammonium ions used in their synthesis.

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Stoichiometric synthesis of a pure ferrite from a tailored layered double hydroxide (hydrotalcite-like) precursor

Junjie Liu, Feng Li, David G. Evans and Xue Duan*



Calcination of a layered double hydroxide precursor with the appropriate ratio of cations [$Mg^{2+}:(Fe^{2+} + Fe^{3+}) = 0.5$] affords a pure ferrite spinel which has a higher saturation magnetization than that produced by conventional routes.

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