

Cover

Cascades featured in the biomimetic syntheses of several members of the bisorbicillinoids, including a double Michael addition/ketalization sequence that generates trichodimerol in a single step, and a Diels–Alder reaction/ring contraction process that gives rise to bisorbibutenolide.



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

contents

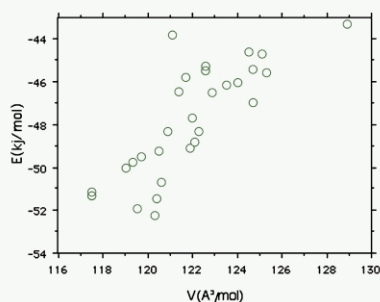
FOCUS ARTICLE

545

Are crystal structures predictable?

Jack D. Dunitz

The one-word answer to the title question is still “No”, although at certain levels of discussion a “Maybe”, or even a conditional “Yes”, may be entertained as possible responses.



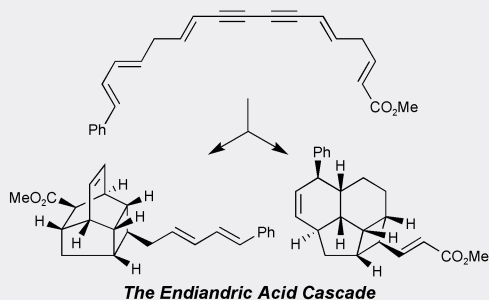
FEATURE ARTICLE

551

Tandem reactions, cascade sequences, and biomimetic strategies in total synthesis

K. C. Nicolaou, Tamsyn Montagnon and Scott A. Snyder

Tandem reactions, cascade sequences, and biomimetic strategies are currently recognized as an ideal means to construct molecular complexity and diversity because of their unparalleled elegance, environmental friendliness, and ability to rapidly generate intricate architectures. This article highlights a number of such processes in the context of natural product total synthesis.



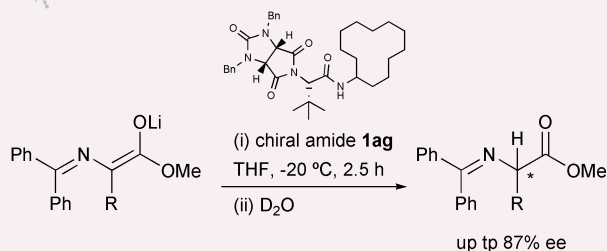
COMMUNICATIONS

566

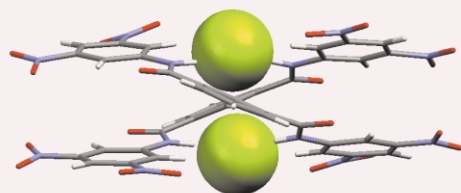
Asymmetric protonation of lithium enolates of α -amino acid derivatives with α -amino acid-based chiral Brønsted acids

Kentaro Futatsugi, Akira Yanagisawa and Hisashi Yamamoto*

A new type of finely designed, chiral α -amino acid-based proton source **1ag** makes it possible to obtain highly enantiopure unnatural α -amino acid derivatives by asymmetric protonation.



568

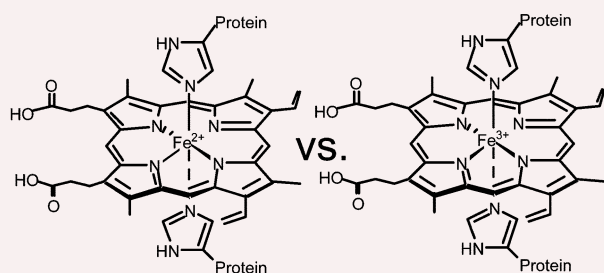


Anion-directed assembly: the first fluoride-directed double helix

Simon J. Coles, Jeremy G. Frey, Philip A. Gale,* Michael B. Hursthouse, Mark E. Light, Korakot Navakhun and Gemma L. Thomas

Two *N,N'*-bis(3,5-dinitrophenyl)isophthalamide molecules wrap around two fluoride anions in the solid state forming the first example of a fluoride directed double helix.

570

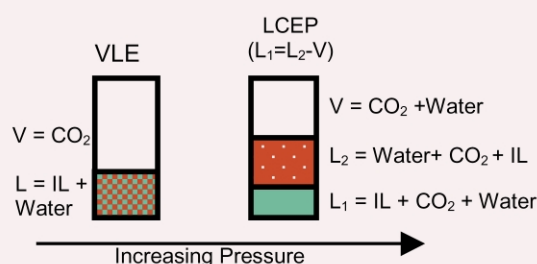


Thermodynamic characterization of ferric and ferrous haem binding to a designed four- α -helix protein

Charles J. Reedy, Michelle L. Kennedy and Brian R. Gibney*

The ferric and ferrous haem binding constants and haem electrochemistry provide insight into the design of a *de novo* designed metalloprotein maquette.

572



Carbon dioxide induced separation of ionic liquids and water

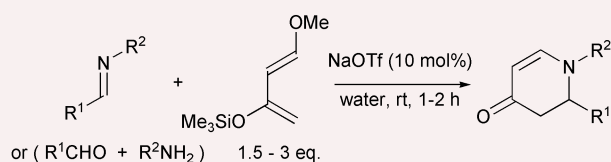
Aaron M. Scurto, Sudhir N. V. K. Aki and Joan F. Brennecke*

A novel environmental technology for the separation of ionic liquids from water using carbon dioxide is demonstrated.

574

Alkaline salt-catalyzed aza Diels–Alder reactions of Danishefsky's diene with imines in water under neutral conditions

Catherine Loncaric, Kei Manabe and Shū Kobayashi*



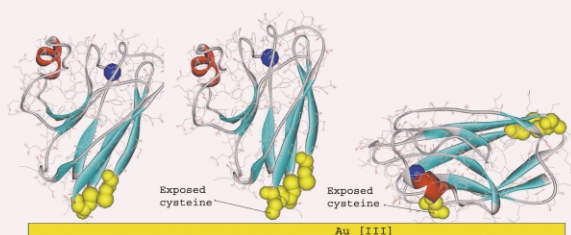
Two- or three-component aza Diels–Alder reactions of Danishefsky's diene with imines or aldehydes and amines in water took place smoothly under neutral conditions in the presence of a catalytic amount of an alkaline salt such as sodium triflate to afford dihydro-4-pyridones in high yields.

576

Genetic modulation of metalloprotein electron transfer at bare gold

Jason J. Davis,* Delphine Bruce, Gerard W. Canters, John Crozier and H. Allen O. Hill

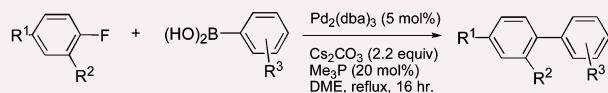
Bioelectrochemical engineering: the electronic coupling between redox-active metalloproteins and electrode surfaces can be engineered by site-directed mutagenesis.



578

Palladium catalysed Suzuki reactions of fluoroarenes

David A. Widdowson* and René Wilhelm

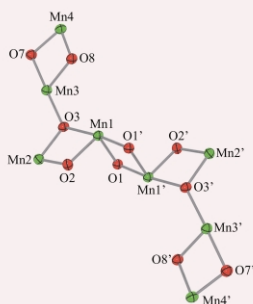


A novel palladium catalysed cleavage of C–F bonds in a series of Suzuki reactions of aryl fluorides is described together with a discussion of the mechanistic implications. This is the first example of uncomplexed fluoroarenes in a palladium catalysed cross-coupling.

580

A novel aggregate of $[\text{Mn}_2(\mu\text{-O})_2]$ units: $[\text{Mn}_8\text{O}_{10}(\text{O}_2\text{CMe})_6(\text{H}_2\text{O})_2(\text{bpy})_6]^{4+}$ with a serpentine core

Anastasios J. Tasiopoulos, Khalil A. Abboud and George Christou*

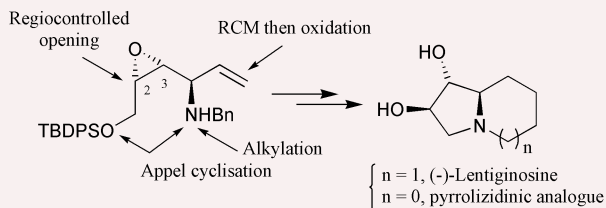


The reaction between $\text{Mn}(\text{O}_2\text{CMe})_2$ and KMnO_4 in aqueous acetic acid in the presence of bpy yields the $6\text{Mn}^{\text{IV}}, 2\text{Mn}^{\text{III}}$ title compound with an unusual serpentine core and an $S = 0$ ground state.

582

Concise asymmetric syntheses of (–)-lentiginosine and of its pyrrolizidinic analogue

Tahar Ayad, Yves Génisson,* Michel Baltas* and Liliane Gorrichon

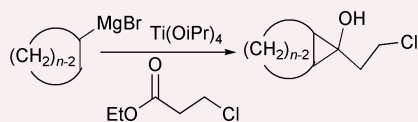


(–)-Lentiginosine and its pyrrolizidinic analogue have been prepared in a straightforward five-step sequence from a versatile chiral *cis*- α,β -epoxyamine.

584

Diastereoselective titanium-mediated construction of *cis*-2,3-ring annelated 1-(2'-chloroethyl)cyclopropanols

Frédéric Lecornué and Jean Ollivier*

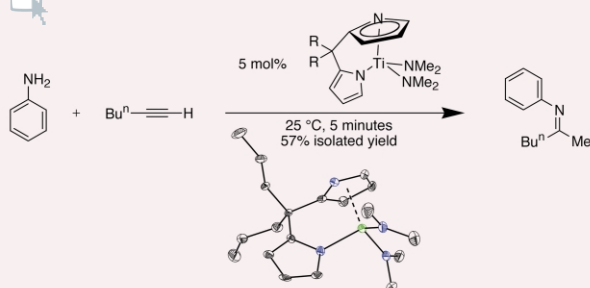


This article presents access to 1-alkyl-2,3-*cis*-fused cyclopropanols in a one-pot reaction and an application to the synthesis of the 8-*exo*-aminobicyclo[5.1.0]octane-8-carboxylic acid.

586

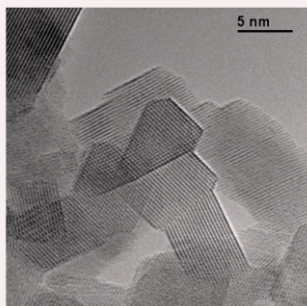
Titanium dipyrrolylmethane derivatives: rapid intermolecular alkyne hydroamination

Yanhui Shi, Christopher Hall, James T. Ciszewski, Changsheng Cao and Aaron L. Odom*



Titanium dipyrrolylmethane complexes are rapid hydroamination catalysts for alkynes with primary amines. Catalysis rate comparisons with other titanium complexes, rates for η^5/η^1 -pyrrolyl exchange, and a short survey of catalytic activity are discussed.

588

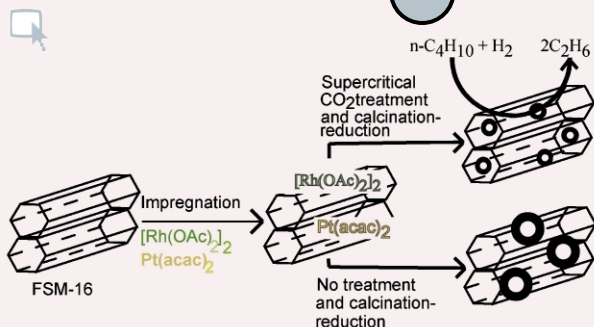


Flame synthesis of nanocrystalline ceria-zirconia: effect of carrier liquid

Wendelin J. Stark, Lutz Mädler, Marek Maciejewski, Sotiris E. Pratsinis* and Alfons Baiker*

The use of carboxylic acid derived carrier liquids in the flame spray synthesis of ceria-zirconia allows production of highly crystalline mixed oxides with improved thermal stability.

590

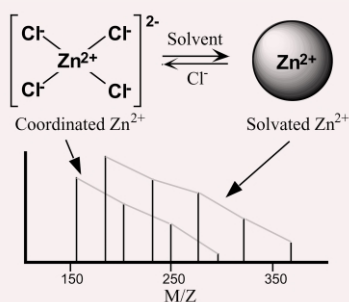


Preparation of highly dispersed RhPt alloy catalysts in mesoporous silica using supercritical carbon dioxide and selective synthesis of ethane in butane hydrogenolysis

Paresh L. Dhepe, Atsushi Fukuoka* and Masaru Ichikawa*

RhPt alloy catalysts were prepared in mesoporous silica using supercritical carbon dioxide in impregnation to achieve high dispersion with controlled morphology; activity and ethane selectivity being enhanced in butane hydrogenolysis.

592

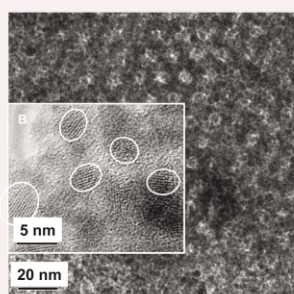


Microscopic environment of metal ion controlled by the balance between preferential solvation and coordination

Shunsuke Mochizuki and Akihiro Wakisaka*

The solvated and coordinated metal ion could be observed directly by mass spectrometric analysis of clusters isolated from liquids droplets.

594

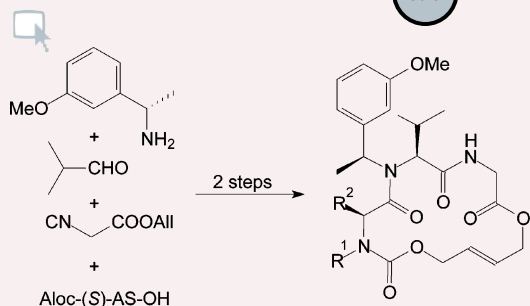


Thermostable sulfated 2–4 nm tetragonal ZrO₂ with high loading in nanotubes of SBA-15: a superior acidic catalytic material

M. V. Landau,* L. Titelman, L. Vradman and P. Wilson

The high-loaded (48–60 wt.%) 2–4 nm tetragonal ZrO₂ phase inserted in mesostructured silica SBA-15 by chemical solution decomposition of Zr(n-PrO)₄ displayed ~3 times higher capacity for surface sulfate ions and, respectively, 1.5–2.2 times higher acidity and catalytic activity in acid-catalyzed reactions compared with bulk SO₄-ZrO₂.

596



Via Ugi reactions to conformationally fixed cyclic peptides

Christina Hebach and Uli Kazmaier

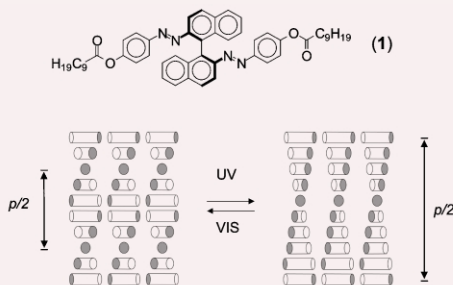
A simple approach to several cyclopeptidomimetics was found *via* the Ugi reaction and subsequent ring-closing metathesis starting from readily available precursors.

598

A new axially-chiral photochemical switch

Silvia Pieraccini, Stefano Masiero, Gian Piero Spada and Giovanni Gottarelli*

Axially chiral molecule **1** undergoes photochemical isomerisation: in this process, both the helical twisting power in nematic liquid crystals and the chiroptical properties are strongly modified: the process can be reversed and the cycle repeated several times without fatigue of the compound.

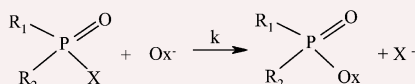


600

The levelling effect of solvational imbalances in the reactions of oximate α -nucleophiles with electrophilic phosphorus centers. Relevance to detoxification of organophosphorus esters

F. Terrier,* E. Le Guével, A. P. Chatrousse, G. Moutiers and E. Buncler*

A study of the reactions of oximate α -nucleophiles with diisopropylphosphorofluoridate (DFP) and two model phosphonates, has revealed either a levelling-off in reactivity or a bell-shaped behaviour in accordance with a critical decoupling of desolvation and bond formation (solvational imbalances); the relevance of these results to detoxification is emphasized.

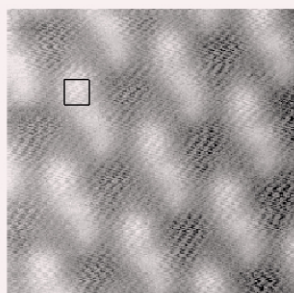


602

Evidence for spatially-coherent trans-molecular electron tunnelling through two-dimensional arrays of Photosystem II core complexes

Philip B. Lukins* and Christopher S. Barton

Scanning tunnelling microscopy is normally believed to image the surface layer of the specimen but we show that trans-molecular electron tunnelling occurs in biological systems and that this tunnelling can be spatially-coherent and localised.

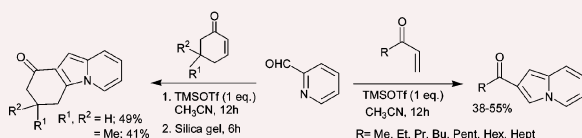


604

First example of electrophile induced Baylis–Hillman reaction: a novel facile one-pot synthesis of indolizine derivatives

Deevi Basavaiah* and Anumolu Jaganmohan Rao

The treatment of pyridine-2-carboxaldehyde with acyclic and cyclic enones in the presence of TMSOTf provides a convenient synthesis of indolizine derivatives in one-pot operation, thus for the first time describing an electrophile induced Baylis–Hillman reaction.

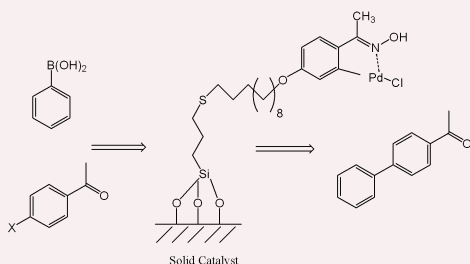


606

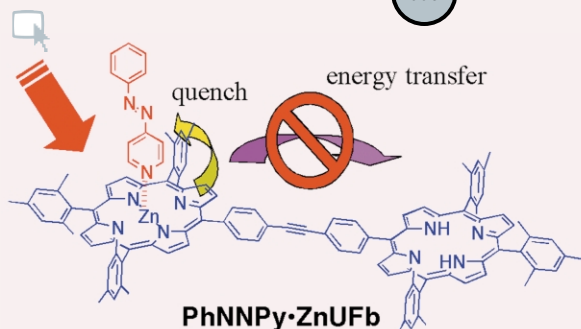
An oxime–carbapalladacycle complex covalently anchored to silica as an active and reusable heterogeneous catalyst for Suzuki cross-coupling in water

Carlos Baleizão, Avelino Corma,* Hermenegildo García* and Antonio Leyva

A preformed carbapalladacycle having an ω -terminated C=C bond eleven C chain anchored to silica is a truly heterogeneous, high-activity, reusable catalyst for Suzuki cross-coupling in water.



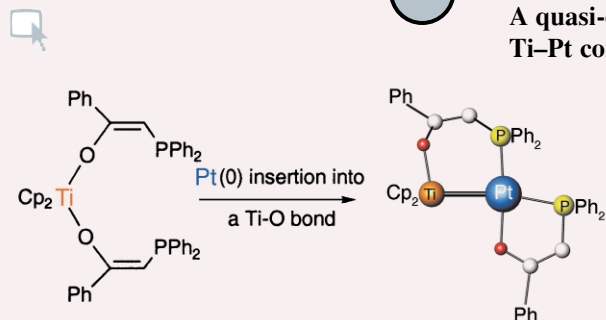
608

**Non-covalent switch for intramolecular energy transfer**

Joe Otsuki,* Akane Yasuda and Toshio Takido

The axially coordinated complex of phenylazopyridine and a Zn-porphyrin/free-base porphyrin conjugate provides a switch for intramolecular energy transfer, with reversible complexation/decomplexation as a switching protocol.

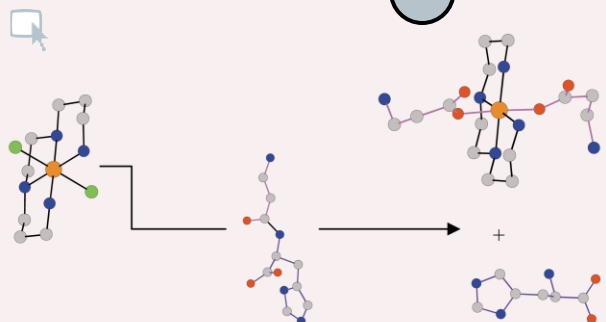
610

**A quasi-covalent metal-metal bond in an early-late heterobimetallic Ti-Pt complex stabilized by phosphinoenolate ligands**

Pierre Braunstein,* Xavier Morise, Marc Bénard, Marie-Madeleine Rohmer and Richard Welter

An unusual early-late bimetallic complex with direct metal-metal bonding Ti(III)-Pt(I) is obtained from a phosphinoenolate ligand-assisted reaction between Ti(IV) and Pt(0) reagents which occurs by formal insertion of the Pt(0) centre into a Ti(IV)-O bond.

612

**An unprecedented *trans*-oriented product from the cleavage of a dipeptide**

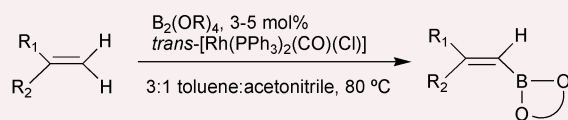
Manas K. Saha and Ivan Bernal*

An unusual *trans* cleavage reaction was observed when *trans*-[Co(3,2,3-tet)Cl₂]Cl {3,2,3-tet = *N,N'*-bis(3-aminopropyl)-ethylenediamine}, was allowed to react with β -alanyl-L-histidine (a bioactive dipeptide) in an aqueous medium at pH \sim 7.5 and 45 °C for 6 h.

614

Rhodium catalysed dehydrogenative borylation of vinylarenes and 1,1-disubstituted alkenes without sacrificial hydrogenation—a route to 1,1-disubstituted vinylboronates

R. Benjamin Coapes, Fabio E. S. Souza, Rhodri Ll. Thomas, Jonathan J. Hall and Todd B. Marder*

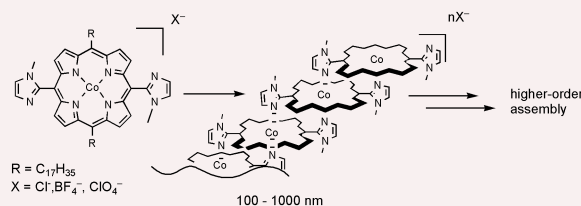


The complex *trans*-[Rh(Cl)(CO)(PPh₃)₂] is an efficient catalyst for the dehydrogenative borylation of 1,1-disubstituted alkenes without consumption of half the alkene substrate by hydrogenation, giving vinyl(boronates) that cannot be made by alkyne hydroboration.

616

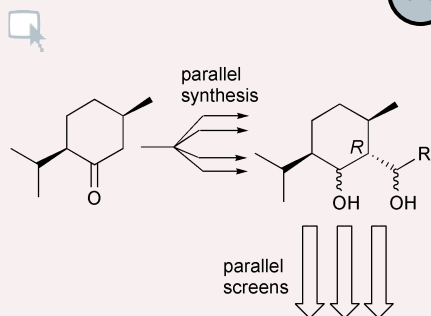
Long rod-like array of bis(imidazolyl)porphyrinatocobalt(III) by successive complementary coordination

Chusaku Ikeda, Eiichi Fujiwara, Akiharu Satake and Yoshiaki Kobuke*



Simple bis(imidazolyl)porphyrin cobalt(III) complex was found to form a long polymeric array by complementary coordination. Formation of higher ordered assemblies was also observed

618

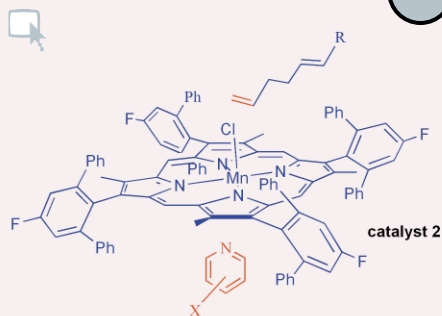


Novel isomenthone-derived 1,3-diol ligands identified through parallel synthesis and screening catalyse an asymmetric aldol reaction

John M. Gardiner,* Philip D. Crewe, Gillian E. Smith and Kenneth T. Veal

A library of new (+)-isomenthone-derived 1,3-diol ligands, containing 5 contiguous stereocentres (3 fixed, 2 variable), was prepared by parallel synthesis. Several ligands from the library catalyse a Mukaiyama aldol reaction with 87–90% e.e.

620

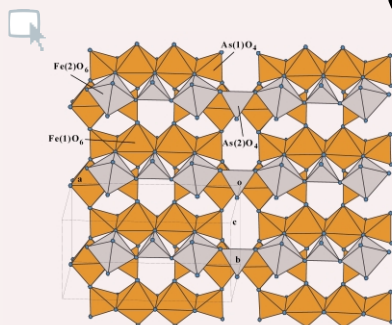


Remarkable axial ligand effect on regioselectivity towards terminal alkenes in epoxidation of dienes by a robust manganese porphyrin

Tat-Shing Lai, Stephen K. S. Lee, Lam-Lung Yeung, Hai-Yang Liu, Ian D. Williams and Chi K. Chang*

The ability of catalyst **2** to structurally regionalizing diene molecules is reinforced by simply adding axial ligand and the selectivity of epoxidation so obtained is complementary to conventional epoxidation reagents.

622

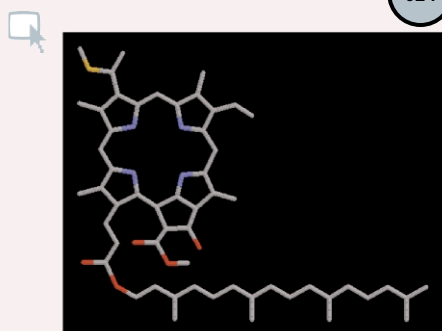


Fe(AsO₄): A new iron(III) arsenate synthesized from thermal treatment of (NH₄)[Fe(AsO₄)F]

Begoña Bazán, José L. Mesa,* José L. Pizarro, Andrés T. Aguayo, María I. Arriortua and Teófilo Rojo*

A new Fe(AsO₄) arsenate has been obtained from thermal treatment of the (NH₄)[Fe(AsO₄)F] precursor maintaining its single-crystalline state.

624

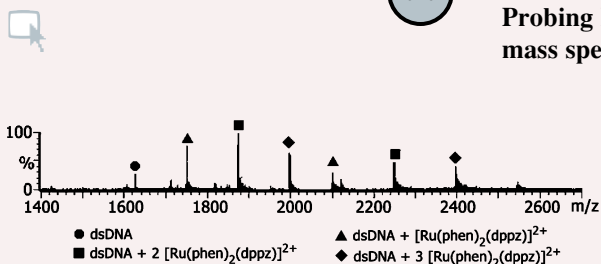


Identification of novel sulfur-containing derivatives of chlorophyll *a* in a Recent sediment

Angela H. Squier, Dominic A. Hodgson and Brendan J. Keely*

Novel transformation products of chlorophyll *a* incorporating a methyl sulfide group in the substituent at the C-3 position have been identified in Recent sediments from an Antarctic lake.

626



Probing DNA selectivity of ruthenium metallointercalators using ESI mass spectrometry

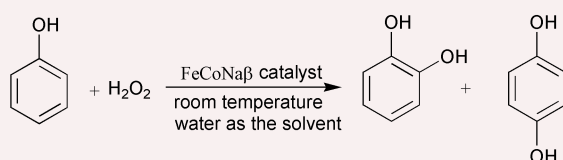
Jennifer L. Beck, Rajesh Gupta, Thitima Urathamakul, Nyree L. Williamson, Margaret M. Sheil, Janice R. Aldrich-Wright and Stephen F. Ralph*

ESI mass spectrometry readily provides information on non-covalent complexes formed between ruthenium complexes and DNA, including relative binding affinities and DNA sequence selectivity.

628

Room-temperature heterogeneous hydroxylation of phenol with hydrogen peroxide over Fe²⁺, Co²⁺ ion-exchanged Na β zeolite

Jun Wang, Jung-Nam Park, Xian-Yong Wei and Chul Wee Lee*

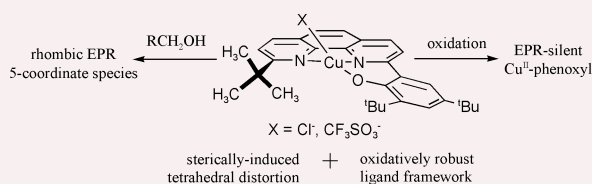


Ion-exchanged Na β zeolite with Fe²⁺ and Co²⁺ cations shows high catalytic activity at room temperature in phenol hydroxylation with H₂O₂, where the conversion of phenol is *ca.* 21% and the selectivity of benzoquinone is below 3% at a molar ratio of phenol to H₂O₂ of 3 in the starting aqueous reaction medium.

630

Oxidatively robust monophenolate-copper(II) complexes as potential models of galactose oxidase

Robertus J. M. Klein Gebbink, Masayuki Watanabe, Russell C. Pratt and T. Daniel P. Stack*

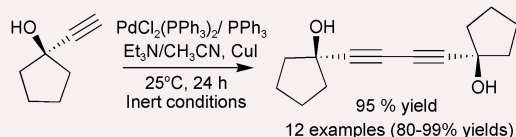


Cupric complexes of a novel phenanthroline-phenolate ligand have distorted coordination geometries and electrochemical properties conducive to modeling the enzyme galactose oxidase.

632

Pd-catalysed cross coupling of terminal alkynes to diynes in the absence of a stoichiometric additive

Ian J. S. Fairlamb,* Patrick S. Bäuerlein, Lester R. Marrison and Julia M. Dickinson

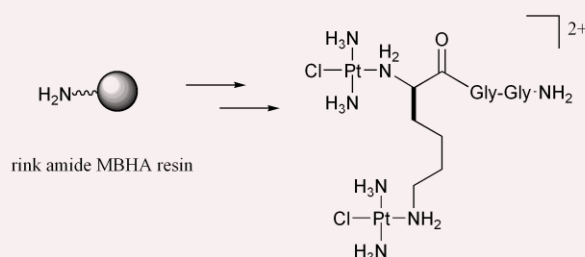


An efficient, room temperature procedure for the cross-coupling of a range of terminal alkynes, using standard Sonogashira cross-coupling conditions (Pd/Cu) is presented. At higher reaction temperatures, head-to-tail or head-to-head dimerisation affords 1,3- and 1,4-disubstituted enynes, respectively as minor products.

634

Extending solid-phase methods in inorganic synthesis: the first dinuclear platinum complex synthesised *via* the solid phase

Steven van Zutphen, Marc S. Robillard, Gijs A. van der Marel, Herman S. Overkleeft, Hans den Dulk, Jaap Brouwer and Jan Reedijk*

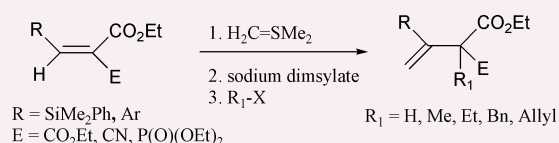


Lysine bridged platinum(II) complexes are synthesised for the first time *via* a solid-phase protocol in an attempt to speed up the discovery of new platinum anticancer agents designed to overcome cisplatin resistance.

636

A novel reaction of dimethylsulfonium methylide with Michael acceptors: application to the synthesis of difficultly accessible vinyl silanes and styrenes

Sunil K. Ghosh,* Rekha Singh and Sonali M. Date

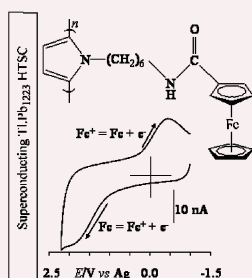


A novel reaction of dimethylsulfonium methylide with activated olefins gave geminally substituted vinyl silanes and styrenes where the loss of hydrogen overrides either the loss of silyl group of cyclopropane formation.

638

Sub- T_c electron transfer at the HTSC/polymer interface

Nicolas Le-Poul, Stephen J. Green* and J. Paul Attfield

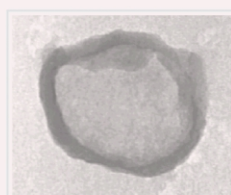
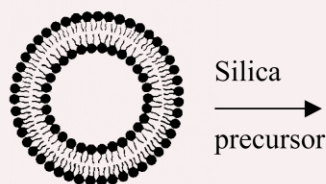


As a novel probe of the superconducting state, the effect of electrode superconductivity has been examined in the first measurements of sub- T_c electron transfer kinetics at the HTSC/polymer interface.

640

Preparation and characterization of siliceous material using liposomes as template

S. Bégu,* R. Durand, D. A. Lerner, C. Charnay, C. Tourné-Péteilh and J. M. Devoisselle

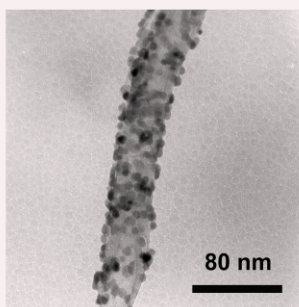


A new synthesis leads to spherical silica particles enclosing their liposomal template. This material insures a high stability to the liposomes and should be a good candidate for a drug delivery system.

642

Decorating catalytic palladium nanoparticles on carbon nanotubes in supercritical carbon dioxide

Xiang R. Ye, Yuehe Lin and Chien M. Wai*

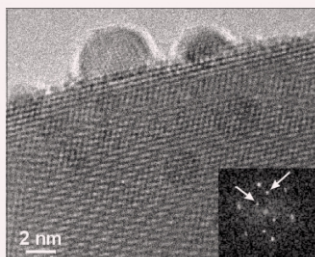
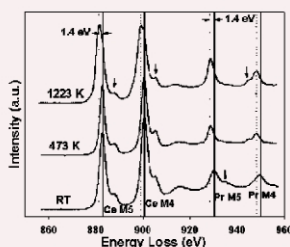


A rapid, direct and clean approach has been developed to decorate catalytic palladium nanoparticles onto multi-walled carbon nanotubes through the simple hydrogen reduction of a Pd(II)- β -diketone complex in supercritical CO_2 .

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***In situ* transmission electron microscopy investigation of Ce(IV) and Pr(IV) reducibility in a Rh (1%)/Ce_{0.8}Pr_{0.2}O_{2-x} catalyst**

C. López-Cartes, S. Bernal, J. J. Calvino,* M. A. Cauqui, G. Blanco, J. A. Pérez-Omil, J. M. Pintado, S. Helveg and P. L. Hansen

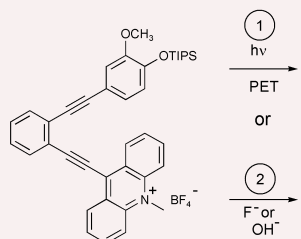


In-situ Atomic Resolution Transmission Electron Microscopy is applied to the challenging problem of investigating simultaneously the redox processes and oxygen vacancy ordering phenomena in ceria-based catalysts containing two reducible cations.

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A novel concept to activate enediynes for DNA cleavage

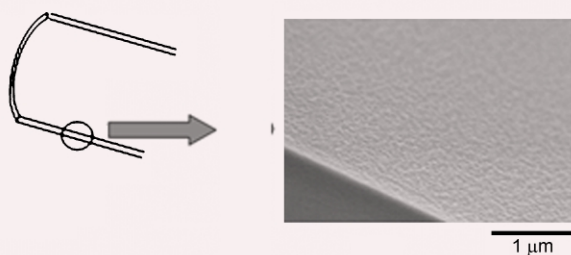
Michael Schmittel,* Giampietro Viola, Francesco Dall'Acqua and Guido Morbach



DNA cleavage

A fundamentally new concept to activate enediynes *via* a biscumulenic intermediate using photoinduced electron transfer (PET) or alternatively (for mechanistic purposes) *via* a thermal desilylation strategy is described. The PET variant allows for efficient long wavelength DNA photocleavage. As such the concept offers an interesting and promising complementary strategy to the well-known mode of action *via* thermal or direct photochemical Bergman cyclisation.

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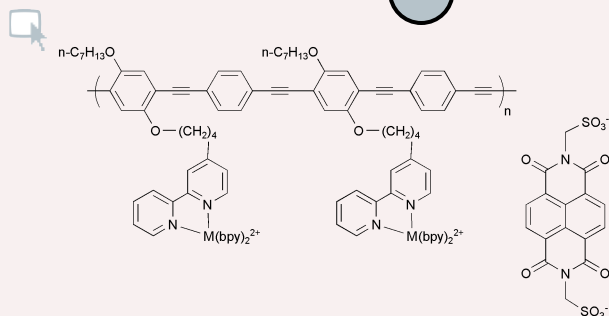


Simple method for preparation of nanostructure on microchannel surface and its usage for enzyme-immobilization

Masaya Miyazaki, Jun Kaneno, Masato Uehara, Masayuki Fujii, Hazime Shimizu and Hideaki Maeda*

We developed a novel preparation method of nanostructure on the microchannel surface, which is suitable for highly efficient enzyme-immobilized microchannel reactor.

650

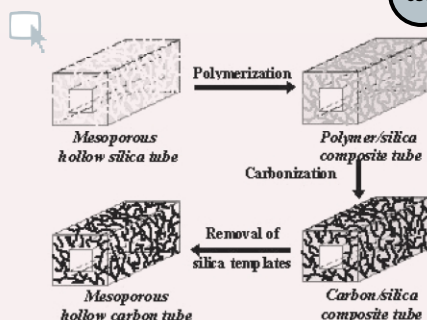


Amplified quenching in metal-organic conjugated polymers

Yao Liu, Shujun Jiang and Kirk S. Schanze*

The luminescence from conjugated polyelectrolytes that contain pendant metal complex units is quenched very efficiently by oppositely charged electron acceptors.

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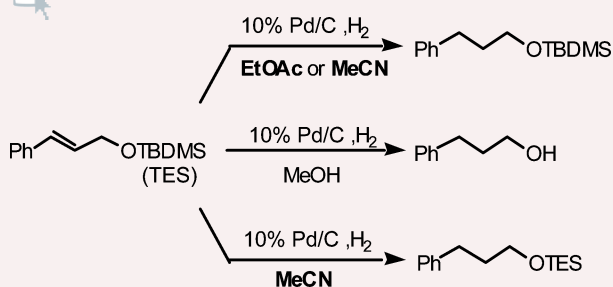


Synthesis of carbon tubes with mesoporous wall structure using designed silica tubes as templates

Minsuk Kim, Kwonnam Sohn, Jaeyun Kim and Taeghwan Hyeon*

Hollow silica tubes with mesoporous walls were synthesized through the sol-gel reaction of tetraethoxysilane and n-octadecyltrimethoxysilane on the surface of *dl*-tartrate self assemblies. Novel hollow carbon tubes with mesoporous walls and rectangular-shaped channels were fabricated using the hollow silica tubes as templates.

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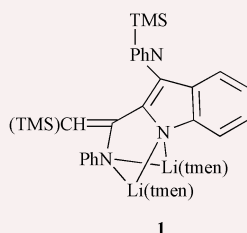


A remarkable solvent effect toward the Pd/C-catalyzed cleavage of silyl ethers

Hironao Sajiki,* Takashi Ikawa, Kazuyuki Hattori and Kosaku Hirota*

Selective hydrogenation conditions of olefin, benzyl ether and acetylene functionalities in the presence of TBDMS or TES ether have been developed.

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1

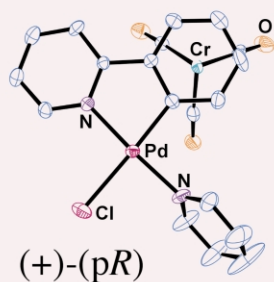
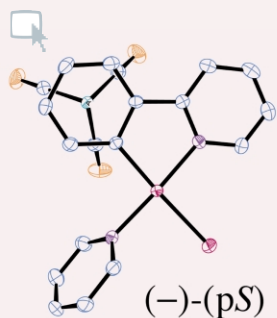
An *N*-lithio-indole from the reaction of $\text{LiCH}(\text{TMS})_2$ and PhNC

Manuel A. Fernandes, Michael F. Lappert, Marcus Layh* and Bernard Omondi

The crystalline *N*-lithio-indole **1** has been obtained in 64% yield from the reaction of equimolar portions of $\text{LiCH}(\text{TMS})_2$, PhNC and $\text{Me}_2\text{NCH}_2\text{CH}_2\text{NMe}_2$ in pentane at low temperature.

COMMUNICATIONS

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Novel heteroleptic *cis*-(C[^]η⁶N)₂Pd(II) chelates for the preparation of enantiopure planar chiral cyclopalladated 2-[tricarbonyl(η⁶-phenyl)chromium]pyridine

Alessandro Berger, Jean-Pierre Djukic,* Michel Pfeffer, André de Cian, Nathalie Kyritsakas-Gruber, Jérôme Lacour and Laurent Vial

Ortho-chloromercurated 2-[(η⁶-phenyl)tricarbonylchromium]pyridine reacts with μ-chloro cyclopalladated aromatic compounds in the presence of [Me₄N]Cl to yield valuable precursors of planar chiral cyclopalladated (η⁶-arene)Cr(CO)₃ complexes.

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