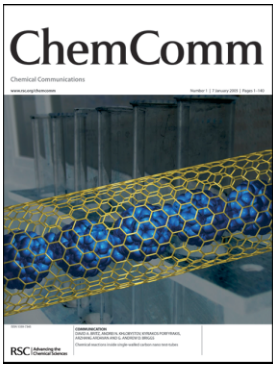
IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (1) 1–140 (2005)

Cover
In 2005, ChemComm celebrates 40 years of successful publication of the highest quality research from across the chemical sciences, with expansion to weekly issues as well as an exciting new look. The cover highlights a space-filling model of a salen-based conducting metallopolymer taken from the Feature Article by Bradley J Holliday and Timothy M Swager. See p. 23.



Inside cover
Computer generated model of a fullerene oxide polymer inside of a carbon nanotube, or so-called "nano test-tube". See p. 37.

CHEMICAL SCIENCE

C1

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

January 2005/Volume 2/Issue 1

www.rsc.org/chemicalscience

EDITORIAL

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ChemComm celebrates 40 years of publishing

Roeland Nolte, Sarah Thomas and Sula Armstrong highlight the exciting developments and new features *ChemComm* is bringing you in this 40th anniversary year.

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FOCUS

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Forty years ago—the first paper

E. W. Meijer

Bert Meijer reviews the career of Hans Wynberg, the author of the first ever *ChemComm* paper back in 1965.

Chemical Communications

13 JANUARY

Synthesis of 2,3,5-Tri-t-butylfuran

FEATURE ARTICLE

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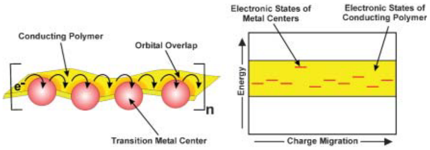
Conducting metallopolymers: the roles of molecular architecture and redox matching

Bradley J. Holliday and Timothy M. Swager*

The role of redox active metals and metal complexes when they are incorporated into conducting polymer systems is reviewed. A highly conductive state, with many potential applications, can be obtained when the two redox active systems are matched through judicious choice of materials. General principles and specific examples are discussed.



COMMUNICATIONS

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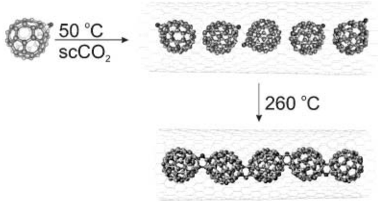
Chemical reactions inside single-walled carbon nano test-tubes

David A. Britz,* Andrei N. Khlobystov,*
Kyriakos Porfyrakis, Arzhang Ardavan and
G. Andrew D. Briggs

SWNTs were used as templates for forming covalent polymeric chains from $C_{60}O$ reacting inside SWNTs; the resulting peapod polymer topology is different from the bulk polymer in that it is linear and unbranched.



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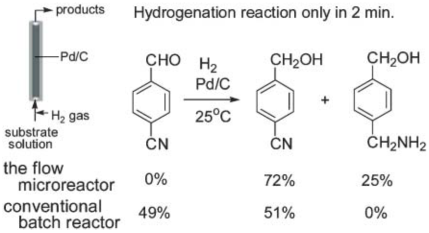
Continuous hydrogenation reactions in a tube reactor packed with Pd/C

Nungruethai Yoswathananont, Kohei Nitta,
Yumi Nishiuchi and Masaaki Sato*

Continuous flow of the substrate solution and hydrogen gas through a microreactor packed with Pd/C brings about a highly reactive and efficient hydrogenation reaction within 2 min.



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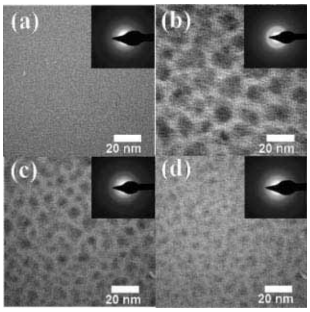

Use of Sn–Si nanocomposite electrodes for Li rechargeable batteries

Hyo-Jin Ahn, Youn-Su Kim, Kyung-Won Park and Tae-Yeon Seong*

Sn nano-dots embedded in Si electrodes are synthesized by a co-sputtering system; their structural properties are correlated to their electrochemical performance during a cycling process.

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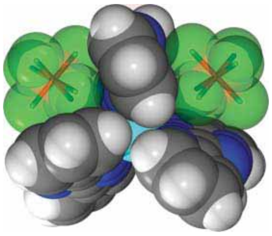

Anion dependence of Ag(I) reactions with 3,6-bis(2-pyridyl)-1,2,4,5-tetrazine (bptz): isolation of the molecular propeller compound $[\mathrm{Ag_2(bptz)_3}][\mathrm{AsF_6}]_2$

Brandi L. Schottel, John Bacsa and Kim R. Dunbar*

The propeller-like cation $[\mathrm{Ag_2(bptz)_3}]^{2+}$, prepared from $\mathrm{AgAsF_6}$ and bptz in $\mathrm{CH_3CN}$, contains three bridging bptz ligands arranged in a *syn* binding mode. The X-ray structure revealed that the $[\mathrm{AsF_6}]^-$ anions pack in the folds of the cation, with the closest contacts being of the type F…tetrazine ring.

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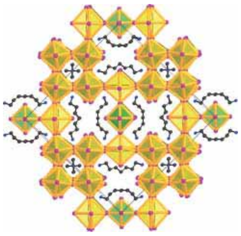

$[\mathrm{H_3N(CH_2)_7NH_3}]_8(\mathrm{CH_3NH_3})_2\mathrm{Sn(IV)Sn(II)}_{12}\mathrm{I}_{46}$ – a mixed-valent hybrid compound with a uniquely templated defect-perovskite structure

Jun Guan, Zhongjia Tang and Arnold M. Guloy*

The semiconducting perovskite-related organic–inorganic hybrid compound $[\mathrm{H_3N(CH_2)_7NH_3}]_8(\mathrm{CH_3NH_3})_2\mathrm{Sn_{13}I_{46}}$ was prepared and characterized; it exhibits a unique defect-perovskite structure and mixed-valent Sn(II/IV).

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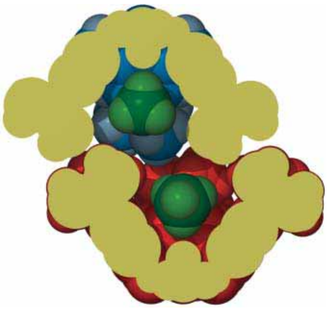

A crystalline organic substrate absorbs methane under STP conditions

Jerry L. Atwood,* Leonard J. Barbour,*
Praveen K. Thallapally and Trevor B. Wirsig

A low density polymorph of the well-known host compound *p-tert*-butylcalix[4]arene absorbs methane at room temperature and atmospheric pressure.

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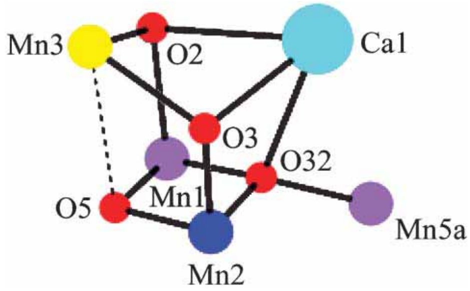
The first high oxidation state manganese–calcium cluster: relevance to the water oxidizing complex of photosynthesis

Abhudaya Mishra, Wolfgang Wernsdorfer, Khalil A. Abboud and George Christou*

The first high oxidation state Mn–Ca cluster has been prepared, mixed-valent $[Mn_{13}Ca_2O_{10}(OH)_2(OMe)_2(O_2CPh)_{18}(H_2O)_4]$. The structure contains $[Mn_4CaO_4]$ sub-units similar to that found in the photosynthetic water oxidizing complex.



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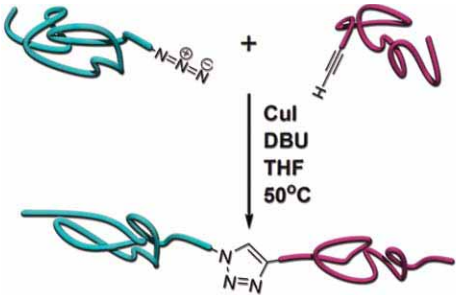
Modular synthesis of block copolymers *via* cycloaddition of terminal azide and alkyne functionalized polymers

Joost A. Opsteen and Jan C. M. van Hest*

Efficient modular synthesis of block copolymers *via* 1,3-dipolar cycloaddition between stable terminal alkyne and azide functionalized polymer blocks, allows full analysis of these building blocks and easy purification of the final product.



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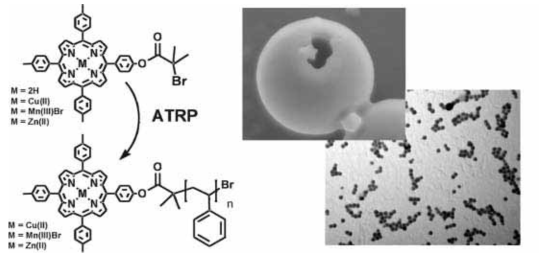
Construction of functional porphyrin polystyrene nanoarchitectures by ATRP

Femke de Loos, Irene C. Reynhout, Jeroen J. L. M. Cornelissen,* Alan E. Rowan* and Roeland J. M. Nolte

A series of novel mono-functionalized metallo-porphyrin polystyrenes have been synthesized using atom transfer radical polymerization. Electron microscopy studies revealed that the resulting amphiphiles self-assemble in water to give functional nanostructures.



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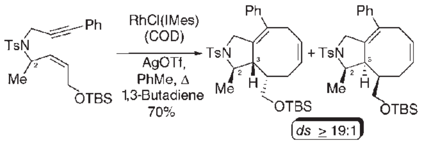
Diastereoselective metal-catalyzed [4 + 2 + 2] carbocyclization reactions utilizing a rhodium *N*-heterocyclic carbene (NHC) complex: the first example of a rhodium NHC-catalyzed [*m* + *n* + *o*] carbocyclization

P. Andrew Evans,* Erich W. Baum, Aleem N. Fazal and Maren Pink

The air and moisture stable rhodium *N*-heterocyclic carbene (NHC) complex, RhCl(IMes)(COD) (IMes = *N*,*N*′-bis(2,4,6-trimethylphenyl)imidazole-2-ylidine; COD = 1,5-cyclooctadiene), facilitates a diastereoselective metal-catalyzed [4 + 2 + 2] carbocyclization of 1,6-enynes in the presence of silver triflate and 1,3-butadiene.



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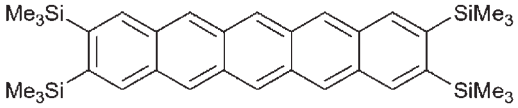

A soluble pentacene: synthesis, EPR and electrochemical studies of 2,3,9,10-tetrakis(trimethylsilyl)pentacene

Siu Hin Chan, Hung Kay Lee, Yu Man Wang, Nan Yan Fu, Xiao Ming Chen, Zong Wei Cai and Henry N. C. Wong*

The title compound is soluble at room temperature in common organic solvents such as hexane, dichloromethane, benzene and methanol. Its chemical and physical properties can easily be studied in solution.

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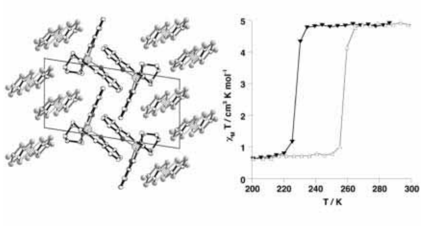

[Fe(sal$_2$-trien)][Ni(dmit)$_2$]: towards switchable spin crossover molecular conductors

Stéphane Dorbes, Lydie Valade, José A. Real* and Christophe Faulmann*

Electrical conductivity and spin transition: a cooperative spin transition with a wide hysteresis loop (30 K) around 240 K has been observed for the first time in an Fe(III) complex, based on the redox active [Ni(dmit)$_2$]$^-$, namely [Fe(sal$_2$-trien)][Ni(dmit)$_2$].

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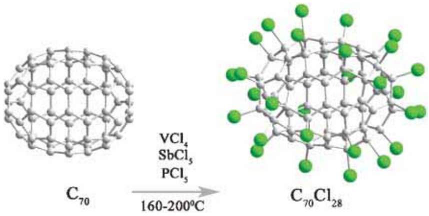

Synthesis and structural characterization of highly chlorinated C$_{70}$, C$_{70}$Cl$_{28}$

Sergey I. Troyanov,* Natalia B. Shustova, Ilya N. Ioffe, Andrew P. Turnbull and Erhard Kemnitz

Chlorination of [70]fullerene with SbCl$_5$, VCl$_4$ or PCl$_5$ yielded C$_{70}$Cl$_{28}$ comprising three isomers, all containing four isolated benzenoid rings in the fullerene cage.

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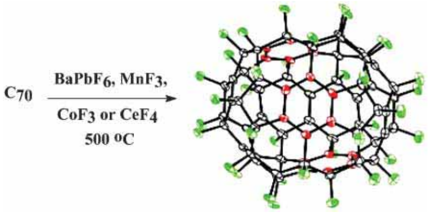

C$_2$ C$_{70}$F$_{38}$ is aromatic, contains three planar hexagons, and has equatorial addends

Peter B. Hitchcock, Anthony G. Avent, Natalia Martsinovich, Pavel A. Troshin and Roger Taylor*

The main isomer of C$_{70}$F$_{38}$ produced on fluorination of [70]fullerene has C_2 symmetry, contains three planar aromatic hexagons (red carbons), and has two fluorines on the equator.

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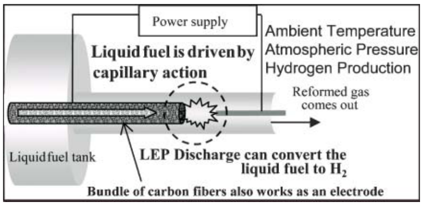
A novel method for hydrogen production from liquid ethanol/water at room temperature

Yasushi Sekine,* Kohei Urasaki, Shinjiro Asai, Masahiko Matsukata, Eiichi Kikuchi and Shigeru Kado

Non-catalytic ethanol steam reforming proceeds efficiently and selectively without coking at the conditions of room temperature and atmospheric pressure using low-energy pulsed (LEP) discharge in combination with carbon fiber electrodes.



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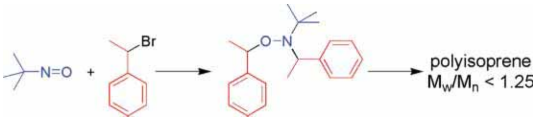
One-step synthesis of alkoxyamines for nitroxide-mediated radical polymerization

Robert B. Grubbs,* Jakub K. Wegrzyn and Qing Xia

An alkoxyamine that is an effective initiator for the controlled polymerization of styrene and isoprene has been prepared by the reaction of 2-methyl-2-nitrosopropane with two equivalents of radicals derived from 1-bromoethylbenzene.



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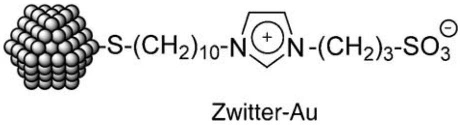
Remarkably stable gold nanoparticles functionalized with a zwitterionic liquid based on imidazolium sulfonate in a high concentration of aqueous electrolyte and ionic liquid

Ryouta Tatumi and Hisashi Fujihara*

Zwitter-Au has great stability in aqueous solutions with high concentrations of electrolytes, ionic liquids, and protein.



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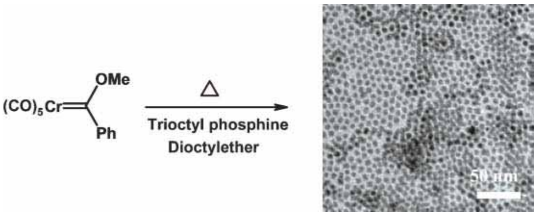
Synthesis of monodisperse chromium nanoparticles from the thermolysis of a Fischer carbene complex

Seung Uk Son, Youngjin Jang, Ki Youl Yoon, Changhua An, Yosun Hwang, Je-Geun Park, Han-Jin Noh, Jae-Young Kim, Jae-Hoon Park and Taeghwan Hyeon*

We successfully synthesized monodisperse chromium nanoparticles using a Fischer carbene complex as precursor.



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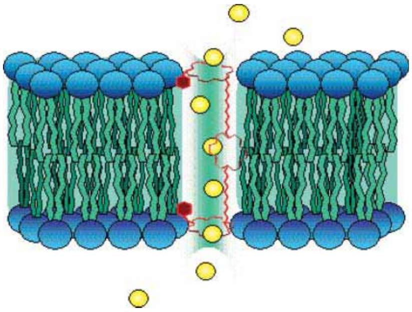

NMR and ion selective electrode studies of hydraphile channels correlate with biological activity in *E. coli* and *B. subtilis*

W. Matthew Leevy, Michelle E. Weber,
Paul H. Schlesinger and George W. Gokel*

Hydraphiles are non-rectifying channels that disrupt osmotic balance when inserted into the membranes of living organisms in proportion to their efficacy *in vitro*.

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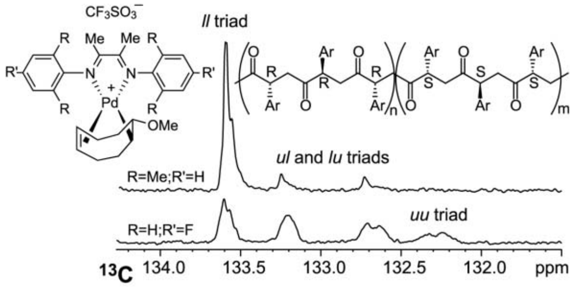

From atactic to isotactic CO/*p*-methylstyrene copolymer by proper modification of Pd(II) catalysts bearing achiral α-diimines

Barbara Binotti, Carla Carfagna,* Cristiano Zuccaccia and Alceo Macchioni*

CO/*p*-methylstyrene copolymers with atactic or isotactic stereoblock structure can be synthesised using Pd(II) catalysts bearing C_{2v}-symmetric α-diimine ligands by a suitable choice of the diimine *ortho*-substituents and counterion.

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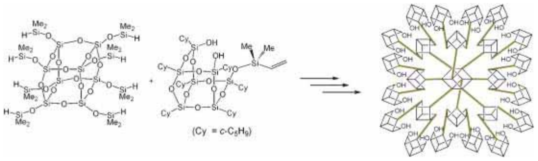

Synthesis of novel starburst and dendritic polyhedral oligosilsesquioxanes

Kenji Wada,* Naoki Watanabe, Koichi Yamada,
Teruyuki Kondo and Take-aki Mitsudo*

Novel starburst-type and second-generation dendritic silsesquioxanes, of which both the core and branched moieties are composed of polyhedral oligosilsesquioxanes, have been synthesized.

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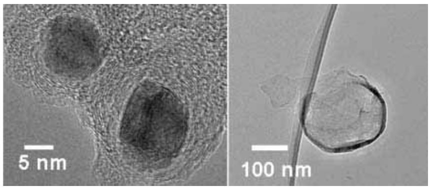

Highly stable carbon-protected cobalt nanoparticles and graphite shells

An-Hui Lu, Wen-Cui Li, Nina Matoussevitch, Bernd Spliethoff, Helmut Bönnemann and Ferdi Schüth*

A synthetic strategy for the fabrication of highly stable carbon-protected cobalt nanoparticles and graphitic hollow shells has been established by using monodispersed cobalt nanoparticles as templates.

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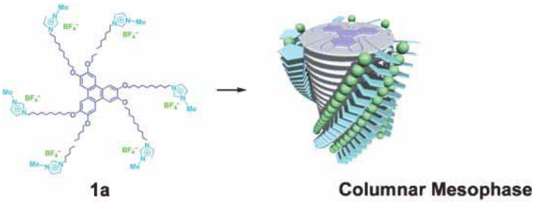
Discotic liquid crystals stabilized by interionic interactions: imidazolium ion-anchored paraffinic triphenylene

Jin Motoyanagi, Takanori Fukushima* and Takuzo Aida*

A triphenylene derivative (**1a**) carrying imidazolium ion-anchored paraffinic side chains forms, over a wide temperature range (111–47 °C), a columnar mesophase, which is retained even at 4 °C when an externally added imidazolium ion-based molten salt is present.



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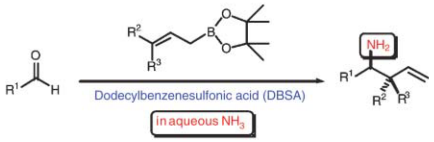
α-Aminoallylation of aldehydes in aqueous ammonia

Shū Kobayashi,* Keiichi Hirano and Masaharu Sugiura

The use of commercial aqueous ammonia in the α-aminoallylation of aldehydes with allylboronates makes the reaction easy and practical.



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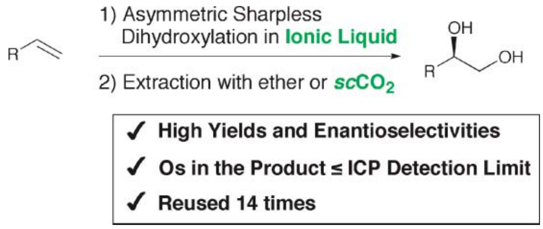
Clean osmium-catalyzed asymmetric dihydroxylation of olefins in ionic liquids and supercritical CO_2 product recovery

Luís C. Branco, Ana Serbanovic, Manuel Nunes da Ponte* and Carlos A. M. Afonso*

The combination of ionic liquids (ILs) as solvents in the asymmetric Sharpless dihydroxylation with the use of *sc*CO_2 in the separation process allows a very simple, efficient, clean and robust system for the reuse of the AD catalytic system.



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Synthesis of cyclopropanes *via* organoiron methodology: preparation of the C9–C16 alkenylcyclopropane segment of ambruticin

Julie M. Lukesh and William A. Donaldson*

A synthesis of the C9–C16 segment of ambruticin is described which relies on organoiron methodology to establish the 1,2,3-trisubstituted cyclopropane ring.

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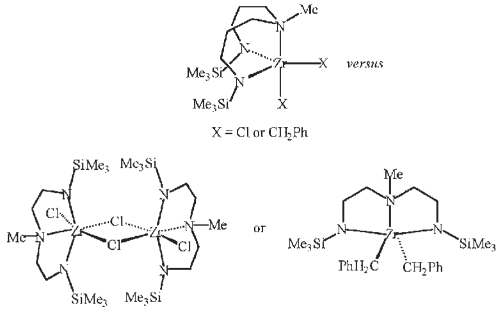

A new diamido-amine ligand based on three-carbon atom "arms": synthesis, structures and polymerisation capability of zirconium derivatives of $MeN(CH_2CH_2CH_2NSiMe_3)_2$

Thomas A. Lowes, Benjamin D. Ward,
Robert A. Whannel, Stuart R. Dubberley and
Philip Mountford*

This contribution reports a new 3-carbon "arm" diamido-amine ligand that is available in multigram quantities from commercially available starting materials, and which exclusively affords *fac* coordination and exhibits promising olefin polymerisation behaviour even with amide-$SiMe_3$ substituents.

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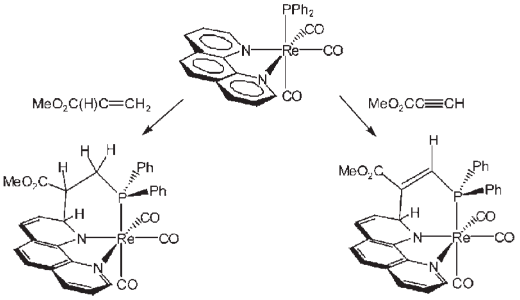

Activation of a 1,10-phenanthroline ligand on a rhenium tricarbonyl complex

Luciano Cuesta, Eva Hevia, Dolores Morales, Julio Pérez,*
Víctor Riera, Elena Rodríguez and Daniel Miguel

Unprecedented activation of ligated 1,10-phenanthroline takes place upon reaction of a rhenium tricarbonyl phosphido complex with an electrophilic alkene or alkyne under mild conditions.

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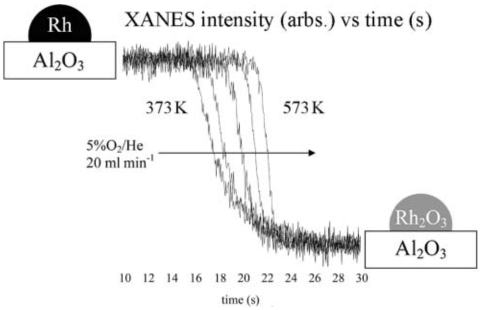

Oxidation/reduction kinetics of supported Rh/Rh_2O_3 nanoparticles in plug flow conditions using dispersive EXAFS

Mark A. Newton,* Steven G. Fiddy, Gemma Guilera,
Bhrat Jyoti and John Evans

Quantitative structural and kinetic parameters for oxidation and reduction of supported Rh/Rh_2O_3 nanoparticles are derived, under plug flow conditions and on a 50 ms time scale, using Rh K edge Energy Dispersive EXAFS (EDE).

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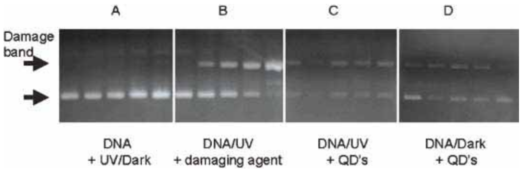

Semiconductor quantum dots and free radical induced DNA nicking

Mark Green* and Emily Howman

The interaction between semiconductor quantum dots and supercoiled DNA is investigated using plasmid nicking assays, suggesting that free radicals, both photogenerated and oxide generated, lead to DNA damage.

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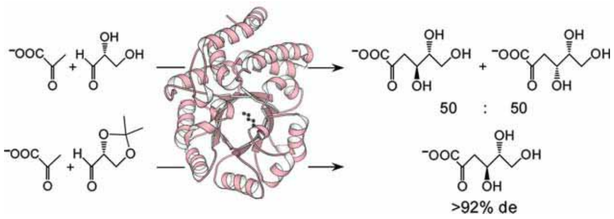
Engineering stereocontrol into an aldolase-catalysed reaction

Henry J. Lamble, Michael J. Danson, David W. Hough and Steven D. Bull*

An extremely thermostable 2-keto-3-deoxygluconate aldolase has been developed for synthetic application. Substrate engineering was used to induce stereocontrol into this naturally-promiscuous enzyme, allowing stereoselective synthesis of one of its own natural substrates.



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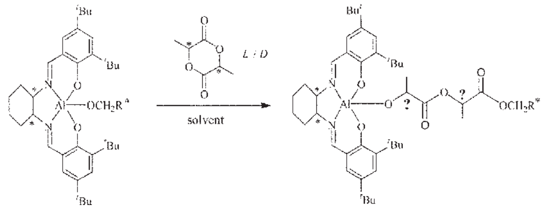
Concerning the relative importance of enantiomorphic site *vs*. chain end control in the stereoselective polymerization of lactides: reactions of (*R*,*R*-salen)- and (*S*,*S*-salen)–aluminium alkoxides LAlOCH$_2$R complexes (R = CH$_3$ and *S*-CHMeCl)

Malcolm H. Chisholm,* Nathan J. Patmore and Zhiping Zhou

The influences of the chirality of the ligand, and initiating end group bound to the metal together with the nature of the solvent, cooperate in a complex manner to determine the diastereoselectivity of the ring opening of lactide.



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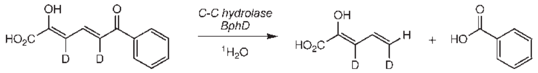
Stereochemistry of the reaction catalysed by 2-hydroxy-6-keto-6-phenyl-hexa-2,4-dienoic acid 5,6-hydrolase (BphD)

Jian-Jun Li and Timothy D. H. Bugg*

The stereochemical course of the reaction catalysed by C-C hydrolase BphD from *Burkholderia xenovorans* LB400 occurs with replacement of a benzoyl group by hydrogen with overall retention of stereochemistry.



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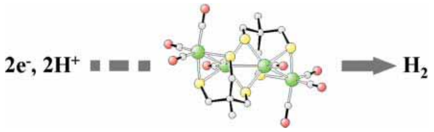
A novel {Fe^I–Fe^{II}–Fe^{II}–Fe^I} iron thiolate carbonyl assembly which electrocatalyses hydrogen evolution

Cédric Tard, Xiaoming Liu, David L. Hughes and Christopher J. Pickett*

Two 'butterfly' {2Fe3S}-cores are fused by two bridging thiolates to form a central planar 2Fe2S unit giving the first example of a chain of four metal–metal bonded iron atoms supported by a bridging sulfur framework: this {Fe^I–Fe^{II}–Fe^{II}–Fe^I} assembly electrocatalyses hydrogen evolution at the all-Fe^I level.



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