ISSN 1359-7345 • CHCOFS (17)1855-2010 (2004)

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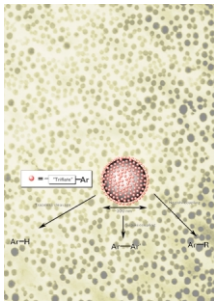

Cover (far left)
Structure of carbamazepine-saccharin against a background photograph of the co-crystals (pp. 1889–1896).

Inside cover (left)
TEM image of surface-functionalised nanobeads which, along with standard polymeric supports, have been elaborated into solid-phase "triflate" equivalents for traceless linking and cross-coupling release strategies (pp. 1914–1915).

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

contents

Chemical Science

September 2004/Volume 1/Issue 9
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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.

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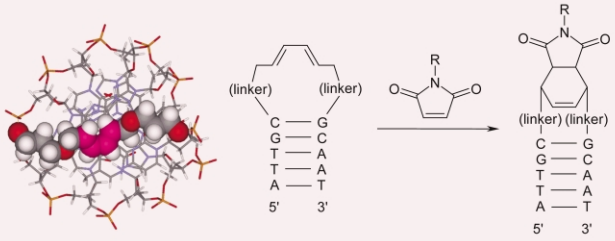
1908

Functionalisation of a diene-modified hairpin mimic *via* the Diels–Alder reaction

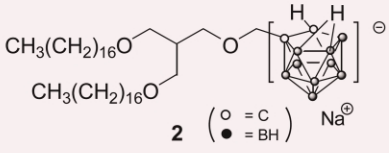


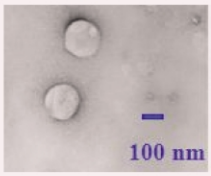
Rolf Tona and Robert Häner*

A highly stable 1,3-butadiene-derived DNA hairpin mimic and its derivatisation *via* the Diels–Alder reaction with various dienophiles are described.

1910

Synthesis and vesicle formation of a nido-carborane cluster lipid for boron neutron capture therapy



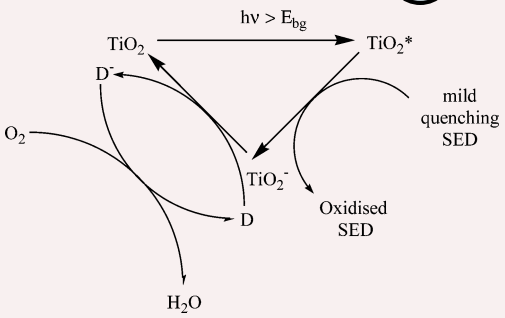


Hiroyuki Nakamura,* Yusuke Miyajima, Toshiaki Takei, Satoshi Kasaoka and Kazuo Maruyama

The nido-carborane lipid **2**, which consists of an ionic boron cluster as a hydrophilic part and a double-tailed moiety, was synthesized for the first time. It was shown by transmission electron microscopy analysis that the lipid **2** formed a stable vesicle and was highly accumulated into DSPC liposomes.

1912

An intelligence ink for oxygen

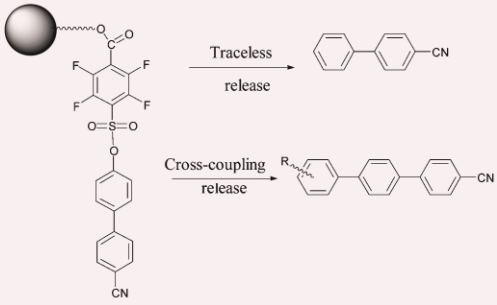


Soo-Keun Lee, Andrew Mills* and Anne Lepre

A novel generic irreversible, UV-activated, re-useable, colorimetric intelligence ink for oxygen comprises semiconductor photocatalyst particles, a brightly-coloured redox dye, a mild reducing agent, a polymer and a solvent.

1914

A novel solid-phase equivalent to the triflate group and its application to traceless linking and cross-coupling-release strategies

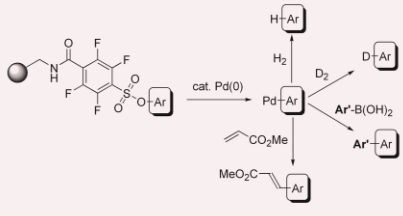


Andrew N. Cammidge* and Zainab Ngaini

A novel polymer solid-phase equivalent to the triflate group has been developed based on polyfluorophenyl sulfonates. The supports can be employed for traceless linking and cross-coupling release strategies.

1916

A 'triflate-like' tetrafluoroarylsulfonate linker for multifunctional solid-phase organic synthesis

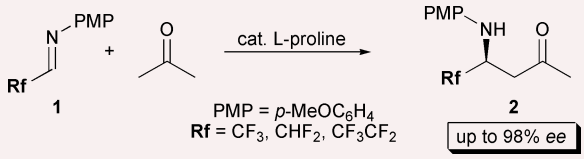


Jefferson D. Revell and A. Ganesan

A new tetrafluoroarylsulfonate solid-phase linker that is readily prepared from pentafluorobenzoic acid is described. The linker functions as a solid-phase 'triflate' equivalent, enabling multifunctional compound cleavage by palladium(0) catalyzed reactions.

First catalytic asymmetric synthesis of β-amino-β-polyfluoroalkyl ketones *via* proline-catalysed direct asymmetric carbon–carbon bond formation reaction of polyfluoroalkylated aldimines

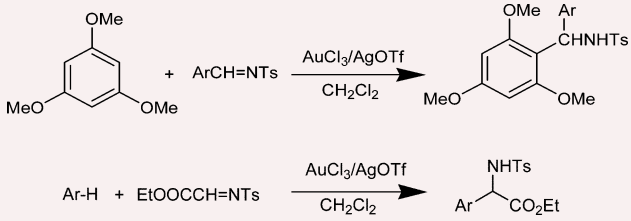
Kazumasa Funabiki,* Masashi Nagamori, Sakiko Goushi and Masaki Matsui



Proline-catalysed direct asymmetric carbon–carbon bond formation reaction of polyfluoroalkylated aldimines with acetone afforded the corresponding β-(*p*-methoxyphenyl)amino-β-polyfluoroalkyl ketones in high enantioselectivities (up to 98% ee).

A highly efficient gold/silver-catalyzed addition of arenes to imines

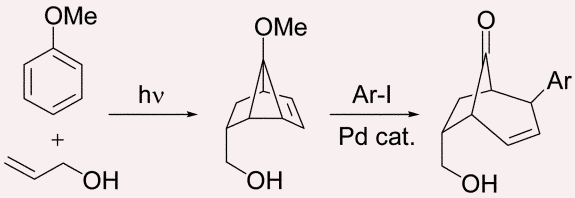
Yumei Luo and Chao-Jun Li*



Various amino acid derivatives are generated.

Palladium catalyzed arylation reactions of *meta* photocycloadducts

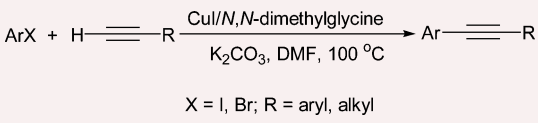
Clive S. Penkett,* Rupert O. Sims, Richard French, Lauriane Dray, Stephen J. Roome and Peter B. Hitchcock



In a highly atom efficient manner a *meta* photocycloaddition reaction between allyl alcohol and anisole followed by a palladium catalyzed Heck reaction provided a bridged bicyclic compound, that modelled the core structure of the alkaloid gelsemine.

CuI-catalyzed coupling reaction of aryl halides with terminal alkynes in the absence of palladium and phosphine

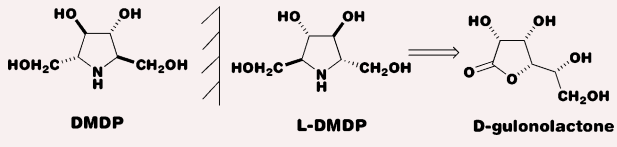
Dawei Ma* and Feng Liu



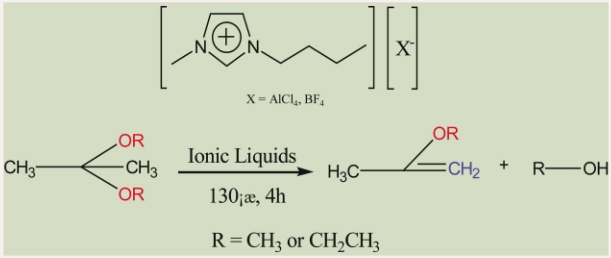
A palladium- and phosphine-free reaction condition for Sonogashira-type coupling was developed, which used CuI/*N*,*N*-dimethylglycine as the catalyst system and was applicable to a range of aryl iodide and aryl bromide substrates.

Looking glass inhibitors: L-DMDP, a more potent and specific inhibitor of α-glucosidases than the enantiomeric natural product DMDP

Chu-Yi Yu, Naoki Asano, Kyoko Ikeda, Mei-Xiang Wang, Terry D. Butters, Mark R. Wormald, Raymond A. Dwek, Ana L. Winters, Robert J. Nash and George W. J. Fleet*



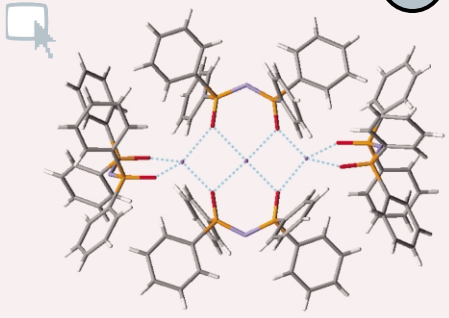
The wrong enantiomer L-DMDP, prepared from D-gulonolactone, is a more specific and potent α-glucosidase inhibitor than the natural sugar mimic DMDP.



Ionic liquids as catalytic green solvents for cracking reactions

Yong Wang, Haoran Li,* Congmin Wang and Hui Jiang

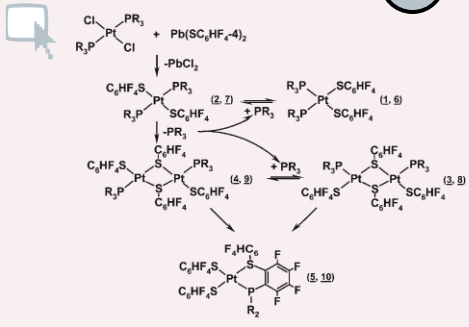
Room-temperature ionic liquids were used as catalysts for the cracking reaction of alkoxypropanes. The elimination reactions of alcohols to give alkenes in ionic liquids are investigated for the first time.



Template-assisted self assembly of two lipophilic polyion aggregates derived from sodium tetraphenyl imidodiphosphinate-complexes containing sodium ions in four different coordination environments

Perla Román-Bravo, Marcela López-Cardoso, Patricia García y García, Herbert Höpfl and Raymundo Cea-Olivares*

The structural organization of alkaline metal ions in ion channels can be simulated by their combination with ligands that mimic the spatial distribution of carbonyl groups in peptides.



Conversion of [Pt(SRf)$_2$(PPh$_{2-n}$(C$_6$F$_5$)$_{n+1}$)$_2$] (*n* = 0 or 1, Rf=C$_6$HF$_4$-4) through carbon–fluorine bond activation to [Pt(SRf)$_2$(1,2-C$_6$F$_4$(SRf)-(PPh$_2$))] and chiral [Pt(SRf)$_2$(1,2-C$_6$F$_4$(SRf)(PPh(C$_6$F$_5$)))]

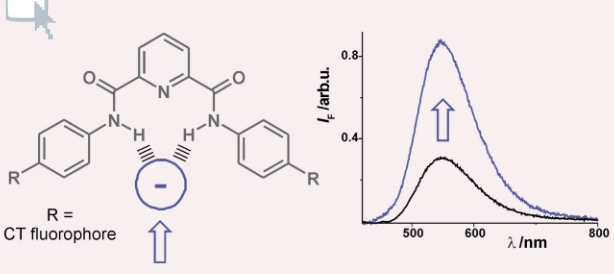
Luis Villanueva, Maribel Arroyo, Sylvain Bernès and Hugo Torrens*

Rare examples of metal promoted C–F activation from PPh$_{3-n}$(C$_6$F$_5$)$_n$ involving polyfluorothiolate ligands to afford 1-thiolate-2-phosphine–tetrafluorophenyl complexes.

Ion/molecule reactions of the protonated serine octamer

Scott Gronert,* Richard A. J. O'Hair* and Adelaide E. Fagin

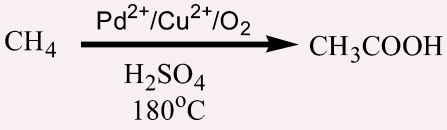
The protonated serine octamer and its derivatives react with amines in the gas phase to give substitution products.



A charge transfer-type fluorescent molecular sensor that "lights up" in the visible upon hydrogen bond-assisted complexation of anions

Anton Kovalchuk, Julia L. Bricks, Günter Reck, Knut Rurack,* Burkhard Schulz, Agnieszka Szumna and Hardy Weißhoff

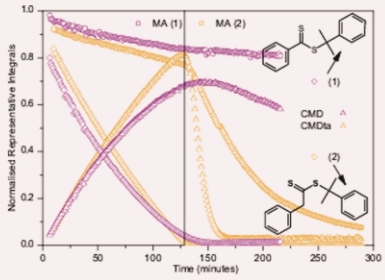
A molecular charge transfer fluorosensor consisting of a bisamidopyridine receptor and two styryl base chromophores shows $H_2PO_4^-$ and acetate-enhanced fluorescence *via* conversion of weak intramolecular into strong intracomplex hydrogen bonds.



Direct oxidation of methane to acetic acid catalyzed by Pd^{2+} and Cu^{2+} in the presence of molecular oxygen

Mark Zerella, Sudip Mukhopadhyay and Alexis T. Bell*

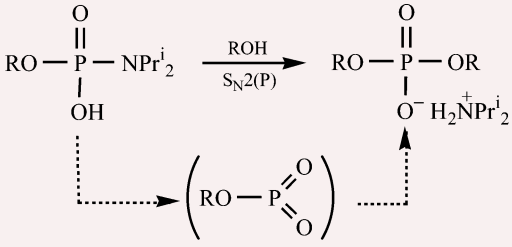
Methane is catalytically converted primarily to acetic acid in concentrated sulfuric acid using a combination of Pd^{2+} and Cu^{2+} in the presence of oxygen. The presence of Cu^{2+} and O_2 enhances the formation of acetic acid and suppresses the reduction of Pd^{2+} to Pd black.



Initialisation in RAFT-mediated polymerisation of methyl acrylate

J. B. McLeary, J. M. McKenzie, M. P. Tonge,* R. D. Sanderson and B. Klumperman*

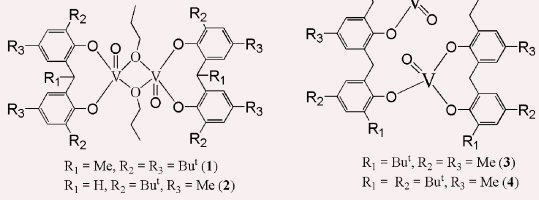
The slow “propagation” of the initiating and leaving group radicals during the early part of methyl acrylate RAFT-mediated polymerisation has characteristics similar to inhibition.



Phosphoramidic acid monoesters as phosphorylating agents: steric effects and reluctance to form monomeric metaphosphate intermediates

Martin J. P. Harger

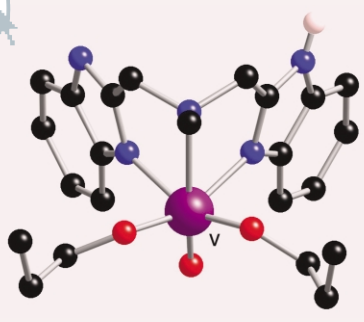
Phosphoramidic acid monoesters cannot readily form monomeric metaphosphate intermediates in the way that their P=S analogues do so they have to react with alcohols by a sterically-sensitive $S_N2(P)$ mechanism.



Vanadyl complexes bearing bi- and triphenolate chelate ligands: highly active ethylene polymerisation procatalysts

Carl Redshaw,* Lee Warford, Sophie H. Dale and Mark R. J. Elsegood

The procatalysts **1–4** show high activity, in combination with dimethylaluminium chloride, for the conversion of ethylene to high molecular weight polyethylene.



Bis(benzimidazole)amine vanadium catalysts for olefin polymerisation and co-polymerisation: thermally robust, single-site catalysts activated by simple alkylaluminium reagents

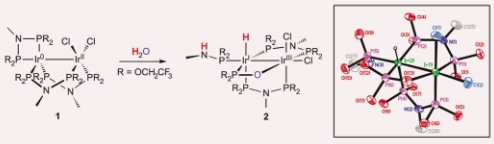
Atanas K. Tomov, Vernon C. Gibson,* Damien Zaher, Mark R. J. Elsegood and Sophie H. Dale

Vanadium complexes containing bis(benzimidazole)amine ligands, when activated by simple alkylaluminium reagents, are found to give highly active, thermally robust, single-site catalysts for olefin polymerisation and co-polymerisation.

1958

Water addition to a two-electron mixed-valence bimetallic center

Adam S. Veige and Daniel G. Nocera*

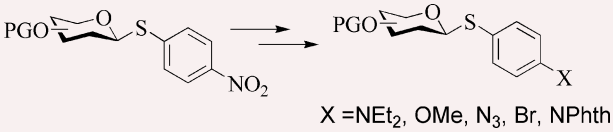


Water reacts at two-electron mixed-valence $Ir^{0,II}_2$ cores ligated by diphosphazane ligands to produce hydride and an unusual dimetal hydroxy phosphite bridge.

1960

One-pot oligosaccharide synthesis: reactivity tuning by post-synthetic modification of aglycon

Lijun Huang, Zhen Wang and Xuefei Huang*

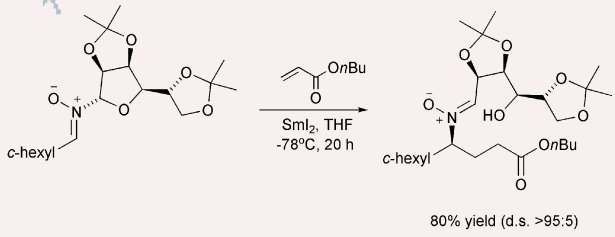


Post-synthetic modification of aglycons produces glycosyl building blocks with multiple levels of anomeric reactivities divergently, which enables one-pot oligosaccharide syntheses.

1962

Radical addition of nitrones to acrylates mediated by SmI_2: asymmetric synthesis of γ-amino acids employing carbohydrate-based chiral auxiliaries

Sine A. Johannesen, Silvia Albu, Rita G. Hazell and Troels Skrydstrup*



An asymmetric version of the samarium diiodide promoted radical addition of aliphatic nitrones to acrylates is disclosed providing examples for the synthesis of γ-amino acid derivatives with high diastereoselectivities (>95 : 5).

1964

Super-hydrophobic tin oxide nanoflowers

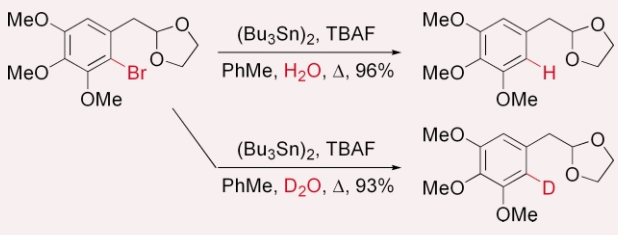
Aicheng Chen,* Xinsheng Peng, Kallum Koczkur and Brad Miller

Super-hydrophobic 3D SnO_2 nanoflowers were fabricated from Sn nanoflowers formed by thermal-pyrolysis of a Sn precursor. The nanoporous SnO_2 flowers possess a large surface area, which supports the potential usefulness of these materials in gas sensor design.

1966

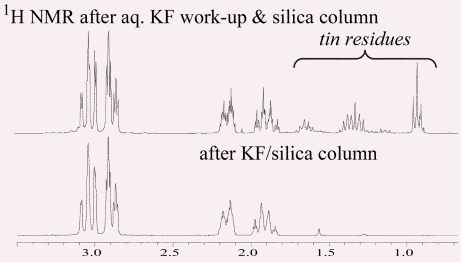
$(Bu_3Sn)_2$–TBAF: a new combination reagent for the reduction and deuteration of aryl bromides and iodides

David C. Harrowven,* Ian L. Guy and Michael I. T. Nunn



$(Bu_3Sn)_2$–TBAF is highly effective for the reduction of aromatic bromides and iodides. When the residual water in TBAF is exchanged for D_2O, the halogen is replaced by a deuterium atom.

1968



KF–Silica as a stationary phase for the chromatographic removal of tin residues from organic compounds

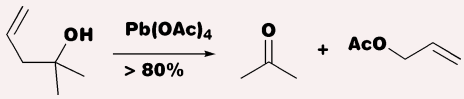
David C. Harrowven* and Ian L. Guy

Through the simple expedient of using a mixture of KF and silica as the stationary phase in column chromatography, levels of organotin impurities from tributyltin hydride mediated reductions have been reduced from stoichiometric levels to ~30 ppm.

1970

A new reaction: lead(IV) acetate-mediated oxidative fragmentation of homoallylic alcohols

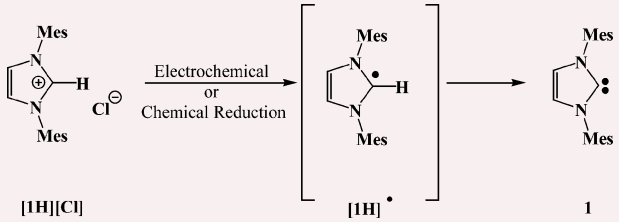
Marcelo D. Preite* and Mauricio A. Cuellar



Treatment of a homoallylic alcohol with lead(IV) acetate results in an oxidative cleavage to give a carbonyl compound and an allylic acetate in good yield, and in stereospecific fashion.

1972

Electrochemical reduction of an imidazolium cation: a convenient preparation of imidazol-2-ylidenes and their observation in an ionic liquid



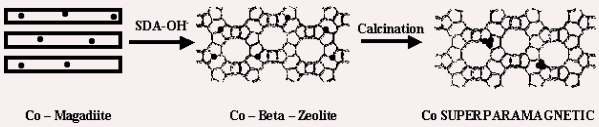
Brian Gorodetsky, Taramatee Ramnial, Neil R. Branda and Jason A. C. Clyburne*

1,3-Bis(2,4,6-trimethylphenyl)imidazolium chloride **[1H][Cl]** is reduced electrochemically and chemically to produce a nucleophilic carbene, namely 1,3-bis(2,4,6-trimethylphenyl)imidazol-2-ylidene **1**. The carbene was also shown to be compatible with and persistent in the ionic liquid tetradecyl(trihexyl)phosphonium chloride.

1974

A new synthetic route to produce metal zeolites with subnanometric magnetic clusters

Eva M. Barea, Vicente Fornés, Avelino Corma,* Patrick Bourges, Emmanuelle Guillon and Victor F. Puntes

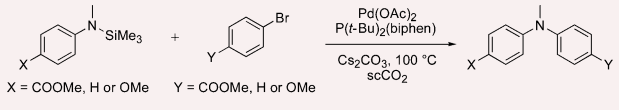


Cobalt in the framework of Beta zeolite becomes superparamagnetic after calcination.

1976

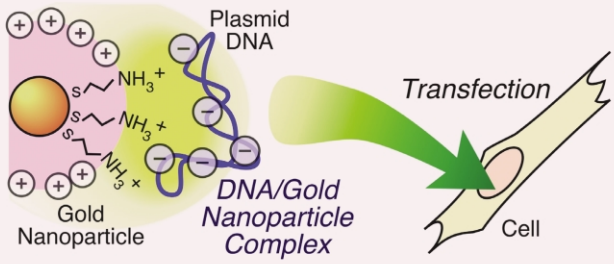
Palladium catalysed cross-coupling reactions of silylamines

Catherine J. Smith,* Tessa R. Early, Andrew B. Holmes* and Richard E. Shute



The palladium-catalysed formation of C–N bonds to produce a range of aryl amines in supercritical carbon dioxide is reported.

1978



Preparation of primary amine-modified gold nanoparticles and their transfection ability into cultivated cells

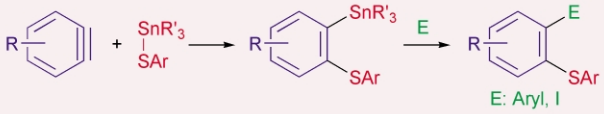
Takuro Niidome,* Kanako Nakashima, Hironobu Takahashi and Yasuro Niidome

Cationic gold nanoparticles, prepared by reducing $HAuCl_4$ with $NaBH_4$ in the presence of 2-aminoethanethiol, formed a complex with DNA, and could be transfected into cells.

1980

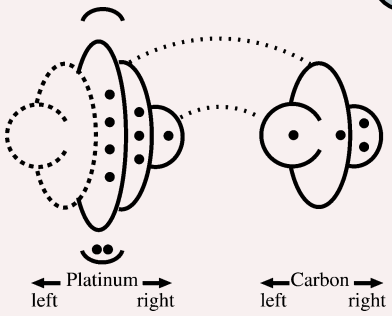
Thiostannylation of arynes with stannyl sulfides: synthesis and reaction of 2-(arylthio)arylstannanes

Hiroto Yoshida,* Tsuguaki Terayama, Joji Ohshita and Atsutaka Kunai*



A sulfur–tin σ-bond of stannyl sulfides was found to add to arynes to afford diverse 2-(arylthio)arylstannanes which were applicable to further transformations.

1982



Darmstadtium carbonyl and carbide resemble platinum carbonyl and carbide

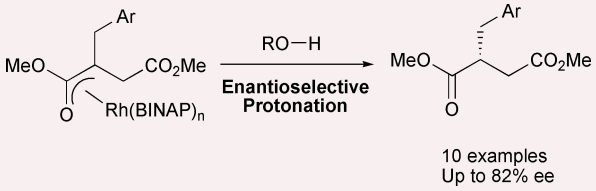
Michael Patzschke and Pekka Pyykkö

DsC and DsCO are calculated to be very similar to PtC and PtCO, suggesting that even the latest 6d elements resemble the corresponding 5d elements chemically.

1984

Rhodium catalysed tandem conjugate addition-protonation: an enantioselective synthesis of 2-substituted succinic esters

Rebecca J. Moss, Kelly J. Wadsworth, Christopher J. Chapman and Christopher G. Frost*

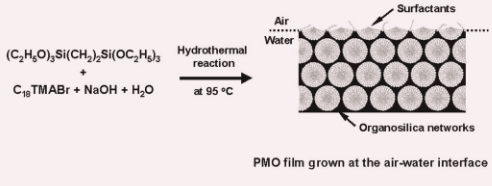


The rhodium catalysed addition of potassium trifluoro(organo)borates to dimethyl itaconate generates an intermediate complex which on protonation provides enantioenriched succinic esters.

1986

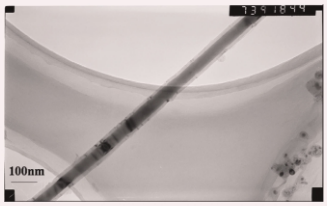
High-quality free-standing and oriented periodic mesoporous organosilica films grown without a solid substrate at the air–water interface

Sung Soo Park and Chang-Sik Ha*



In this communication, we report the first synthesis of high-quality free-standing and oriented periodic mesoporous organosilica (PMO) films grown without a solid substrate by surfactant templating at the air–water interface.

1988

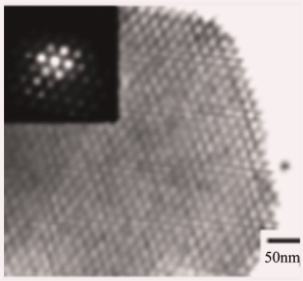


Super-long continuous Ni nanowires encapsulated in carbon nanotubes

Lunhui Guan, Zujin Shi, Huanjun Li, Liping You and Zhennan Gu*

Super-long continuous Ni-filled carbon nanotubes were prepared by the chemical vapor deposition method with cloth-like single-walled carbon nanotube raw soot produced by the arc-discharge method as catalyst.

1990

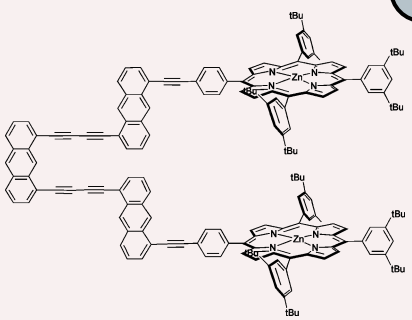


A highly efficient heterogeneous catalytic system for Heck reactions with a palladium colloid layer reduced *in situ* in the channel of mesoporous silica materials

Liang Li, Jian-lin Shi* and Ji-na Yan

The synthesis and characterization of a palladium colloid layer grafted mesoporous SBA-15 material, designated Pd–SBA, are described; the ultrahigh specific area, the large pore opening, and the highly dispersed catalyst species in the Pd–SBA material create one of the most active heterogeneous catalysts for Heck coupling reactions.

1992

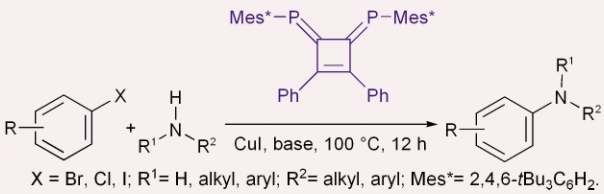


Adjustable cavity for host–guest recognition in cofacial bis-porphyrinic tweezer

Régis Rein, Maurice Gross and Nathalie Solladié*

The synthesis of a cofacial bis-porphyrinic tweezer bearing a tris-anthracenic spacer is reported. Its behavior as host has been evidenced as well as the ability of its cavity to adjust to guests of various sizes.

1994

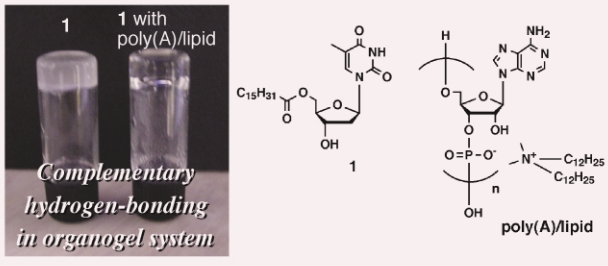


Application of a diphosphinidenecyclobutene ligand in the solvent-free copper-catalysed amination reactions of aryl halides

Anil S. Gajare, Kozo Toyota, Masaaki Yoshifuji* and Fumiyuki Ozawa

Copper-catalysed amination reactions of halobenzenes with amines proceed at 100 °C in the presence of a diphosphinidenecyclobutene (2 mol%), CuI (2 mol%), and *t*-BuOK without solvent, providing an efficient method for the introduction of one or two aryl groups into amines.

1996

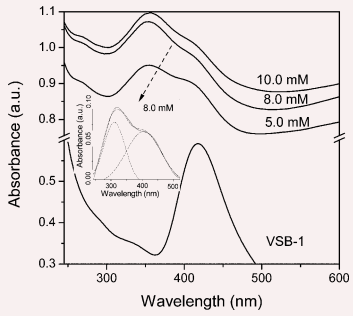


Complementary hydrogen-bonding between thymidine-based low molecular-weight gelator and polynucleotide in organic media

Kazunori Sugiyasu, Munenori Numata, Norifumi Fujita, Sun Min Park, Young Ji Yun, Byeang Hyean Kim and Seiji Shinkai*

Aggregation mode and gelation property of thymidine-based organogelator **1** are affected by the addition of its complementary polynucleotide in organic media.

1998



Preparation and properties of an ordered, uniform 0.9 nm Ag array assembled in a nanoporous VSB-1 by a simple soft chemical method

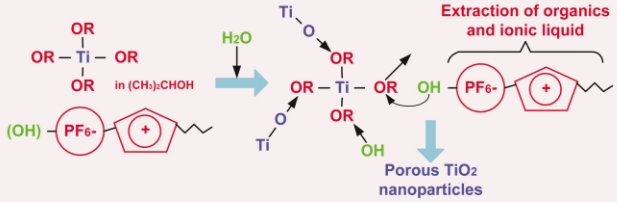
Zhi Chen, Qiuming Gao,* Chundong Wu, Meiling Ruan and Jian-lin Shi

An ordered and uniform 0.9 nm Ag array, exhibiting an unusual UV–vis absorption and controllable intensity, has been synthesized in the channels of a nanoporous VSB-1 by a simple chemical method, presenting potential applications in the theoretical research and in the fabrication of future quantum devices.

2000

Ionic liquid assisted preparation of nanostructured TiO_2 particles

Kyesang Yoo, Hyeok Choi and Dionysios D. Dionysiou*



Anatase-containing nanostructured TiO_2 particles with high surface area and tailor-designed pore size distribution prepared by self-assembled ionic liquid assisted sol–gel methods at low temperature.

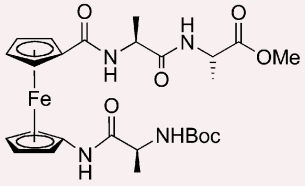
2002

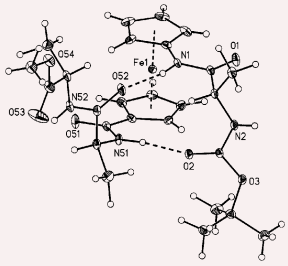
α,β-Unsaturated imines from titanium hydroamination and functionalization by rhodium C–H activation

Changsheng Cao, Yahong Li, Yanhui Shi and Aaron L. Odom*

Titanium pyrrolyl complexes are effective catalysts for the synthesis of α,β-unsaturated imines, which can be further functionalized using Rh-catalyzed alkene or alkyne insertion into the β-C–H bond.

2004



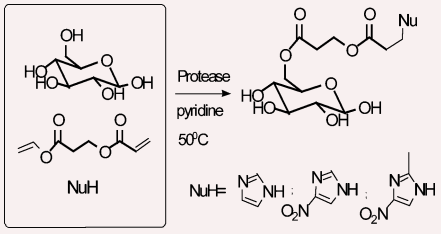


The first oligopeptide derivative of 1′-aminoferrocene-1-carboxylic acid shows helical chirality with antiparallel strands

Lidija Barišić, Maja Dropučić, Vladimir Rapić,* Hans Pritzkow, Srećko I. Kirin and Nils Metzler-Nolte*

The first oligopeptide of the organometallic amino acid 1′-aminoferrocene-1-carboxylic acid shows very stable intra-molecular hydrogen bonding between *anti-parallel* peptide strands in the solid state and in solution.

2006



A single-enzyme, two-step, one-pot synthesis of *N*-substituted imidazole derivatives containing a glucose branch *via* combined acylation/Michael addition reaction

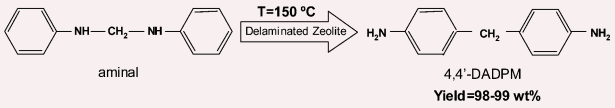
Shi-Ping Yao, De-Shui Lu, Qi Wu, Ying Cai, Shan-Hao Xu and Xian-Fu Lin*

Combined regioselective acylation/Michael addition catalyzed by alkaline protease from *Bacillus subtilis* in pyridine for synthesis of *N*-substituted imidazole derivatives containing a glucose branch *via* a novel single-enzyme, two-step, one-pot procedure is reported.

2008

Replacing HCl by solid acids in industrial processes: synthesis of diamino diphenyl methane (DADPM) for producing polyurethanes

Avelino Corma,* Pablo Botella and Chris Mitchell



Delaminated zeolites show high activity and long catalyst life in the synthesis of diamino diphenyl methane (DADPM), the intermediate in the production of polyurethanes.

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