

**Cover**  
The halogens and the catalytic chemistry behind asymmetric electrophilic halogenation. Illustration kindly provided by Reto Togni.

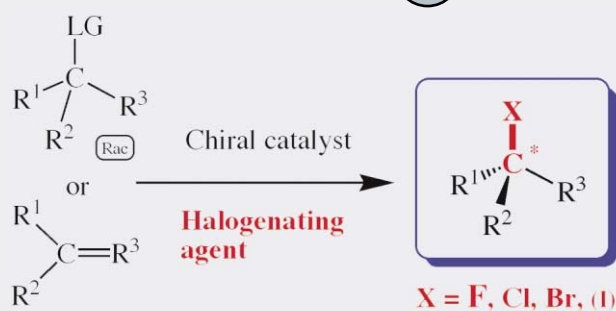


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# contents

## FEATURE ARTICLE

1147



### Enantioselective halogenation reactions

Hasim Ibrahim\* and Antonio Togni\*

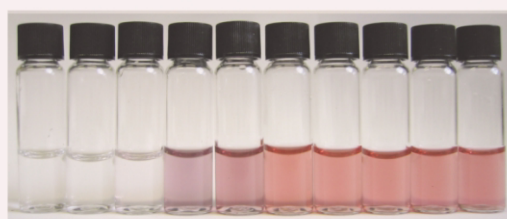
Recent progresses in the field of stereoselective halogenation chemistry, with particular emphasis on still rare transition-metal-catalysed reactions, are reviewed and discussed.

## COMMUNICATIONS

1156



Number of Biotinylated PDP Dextrans per Nanoparticle  
0 0.2 0.4 0.6 0.7 0.8 1.0 1.2 1.4 1.6

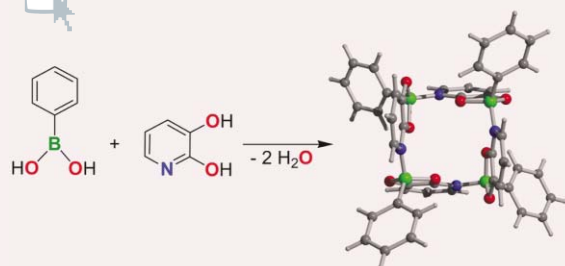


### One molecule per particle method for functionalising nanoparticles

Robert Wilson,\* Yang Chen and Jenny Aveyard

Gold nanoparticles, with a diameter of 15 nm, are stabilised by a minimum of one molecule of 2000 kDa biotinylated PDP dextran per particle.

1158



### A new method for the synthesis of boronate macrocycles

Nicolas Christinat, Rosario Scopelliti and Kay Severin\*

The condensation of aryl boronic acids with 2,3-dihydropyridine gives boronates, which self-assemble to form tetrameric macrocycles as evidenced by X-ray crystallographic analyses.

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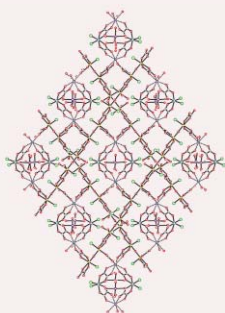
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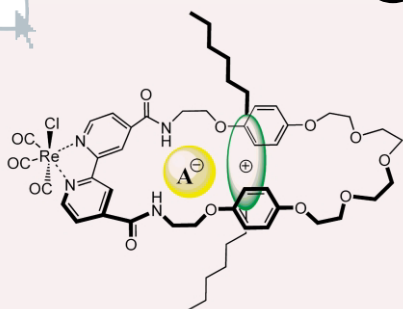


### A novel supramolecular assembly in an iron(III) compound exhibiting magnetic ordering at 70 K

Donatella Armentano, Giovanni De Munno,\* Teresa F. Mastropietro, Miguel Julve\* and Francesc Lloret

The new three-dimensional iron(III) compound of formula  $\{[\text{EtNH}_3][\text{Fe}(\text{H}_2\text{O})_6]\text{Fe}(\text{III})[\text{Fe}_2\text{O}(\text{ox})_3\text{Cl}_2][\text{Fe}(\text{ox})_2\text{Cl}_2]\}_n \cdot 6n\text{H}_2\text{O}$  contains four crystallographically independent iron(III) units,  $[\text{Fe}(\text{ox})_2\text{Cl}_2]^{3-}$ ,  $[\text{Fe}_2\text{O}(\text{ox})_3\text{Cl}_2]^{4-}$ ,  $\text{Fe}^{3+}$  and  $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$  interacting with each other to afford an unprecedented supramolecular three-dimensional network. The compound exhibits magnetic ordering with  $T_c = 70$  K due to a *weak spin canting*.

1162

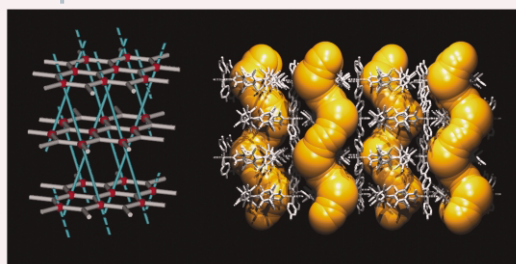


### Halide anion directed assembly of luminescent pseudorotaxanes

David Curiel, Paul D. Beer,\* Rowena L. Paul, Andrew Cowley, Mark R. Sambrook and Fridrich Szemes

A series of new photo-active rhenium(I) bipyridyl based pseudorotaxanes complexes is assembled *via* halide anion templation.

1164

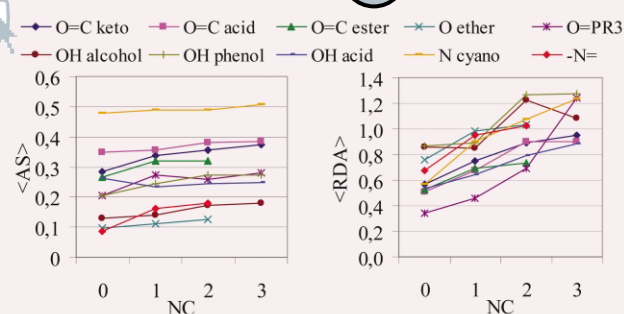


### A new (6<sup>3</sup>)-(6<sup>9</sup>.8<sup>1</sup>) non-interpenetrated paramagnetic network with helical nanochannels based on a tricarboxylic perchlorotriphenylmethyl radical

Daniel Maspoch, Daniel Ruiz-Molina, Klaus Wurst, Concepció Rovira and Jaume Veciana\*

Reaction of a 3-connecting tricarboxylic perchlorotriphenylmethyl radical and a 2-connecting 4,4'-bipyridine ligands with  $\text{Co}(\text{MeCOO})_2 \cdot 4\text{H}_2\text{O}$  forms a paramagnetic non-interpenetrated supramolecular network,  $\text{Co}(\text{PTMTC})(4,4'\text{-bpy})(\text{H}_2\text{O})_3$  (MOROF-2), with an unprecedented (6<sup>3</sup>)-(6<sup>9</sup>.8<sup>1</sup>) topology exhibiting helical nanochannels.

1166

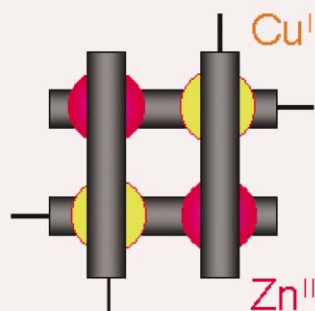


### The probable number of hydrogen-bonded contacts for chemical groups in organic crystal structures

Lourdes Infantes\* and W. D. Sam Motherwell

A crystal structure database of intermolecular contacts has shown that the number of hydrogen bonded contacts increases with average sterically accessible atom surface, <AS>, and with increasing donor/acceptor ratio, <RDA>.

1168



### Programmed single step self-assembly of a [2 × 2] grid architecture built on metallic centers of different coordination geometries

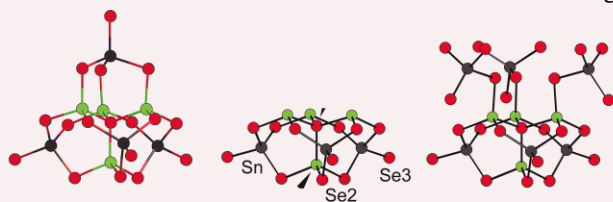
Anne Petitjean, Nathalie Kyritsakas and Jean-Marie Lehn\*

A suitably designed ligand allows the site selective, single step self-assembly of a grid type array containing two tetrahedral Cu(I) and two octahedral Zn(II) metal ion coordination centres.

1170

**K<sub>6</sub>Cd<sub>4</sub>Sn<sub>3</sub>Se<sub>13</sub>: A polar open-framework compound based on the partially destroyed supertetrahedral [Cd<sub>4</sub>Sn<sub>4</sub>Se<sub>17</sub>]<sup>10-</sup> cluster**

Nan Ding, Duck-Young Chung and Mercuri G. Kanatzidis\*

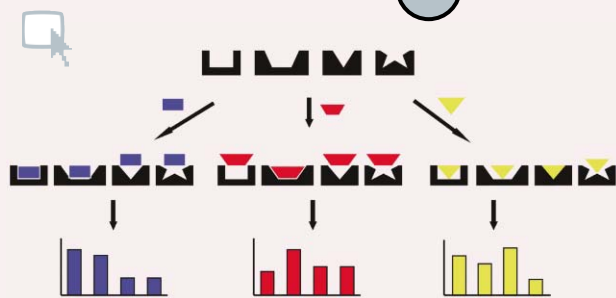


The compound K<sub>6</sub>Cd<sub>4</sub>Sn<sub>3</sub>Se<sub>13</sub> represents an example of a chalcogenide open-framework with the polonium topology.

1172

**Molecularly imprinted polymer sensor arrays**

Nathaniel T. Greene, Stephen L. Morgan and Ken D. Shimizu\*

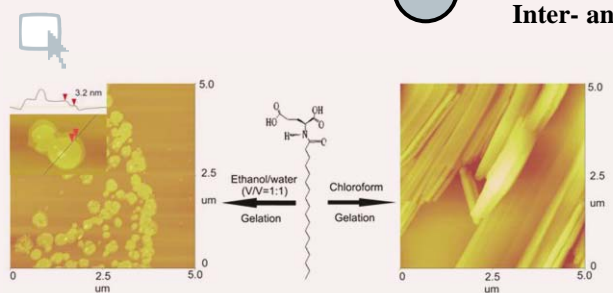


Utilizing molecular imprinted polymers (MIPs) in an array format yields unique patterns in the presence of seven aryl amine analytes, enabling their classification with a high degree of accuracy.

1174

**Inter- and intra-molecular H-bonds induced different nanostructures from a multi-H-bonding (MHB) amphiphile: nanofibers and nanodisks**

Peng Gao, Chuanlang Zhan,\* Lizhu Liu, Yanbiao Zhou and Minghua Liu\*

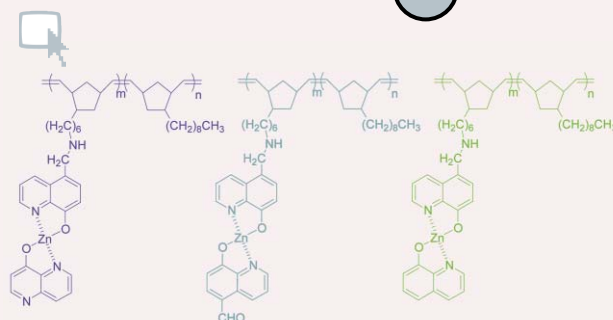


Inter- and intra-molecular H-bonds induced different nanostructures for *N*-stearoyl-*L*-glutamic acid, an multi-H-bonding amphiphile, in different solvents, which were confirmed by XRD, FT-IR and temperature dependent <sup>1</sup>H-NMR data.

1176

**Design, synthesis, characterization, and fluorescent studies of the first zinc-quinolate polymer**

Amy Meyers, Clint South and Marcus Weck\*

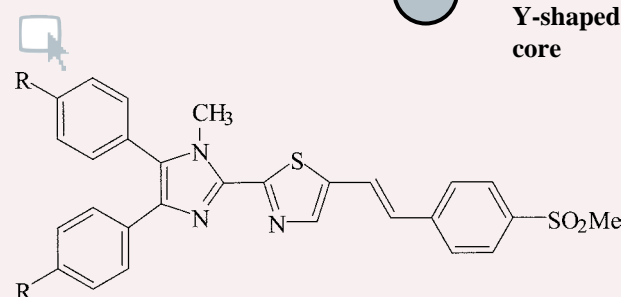


The synthesis and characterization of the first Znq<sub>2</sub>-monomers yielded polymers and copolymers with the desired photoluminescence properties of Znq<sub>2</sub>, emission colors ranging from blue to yellow, while maintaining solution processability of a polymer.

1178

**Y-shaped two-photon absorbing molecules with an imidazole-thiazole core**

Ke Feng, Leonardo De Boni, Lino Misoguti, C. R. Mendonça, Michael Meador, Fu-Lian Hsu and Xiu R. Bu\*



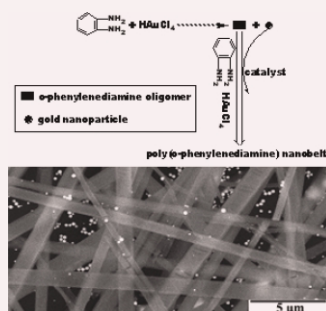
Two new classes of two-photon absorbing Y-shaped molecules have been developed to possess an imidazole-thiazole core and a stilbene-type conjugation pathway with either nitro or sulfonyl as terminal electron-accepting group.



1182

### Large scale, templateless, surfactantless route to rapid synthesis of uniform poly(*o*-phenylenediamine) nanobelts

Xuping Sun, Shaojun Dong\* and Erkang Wang\*

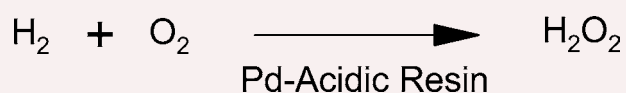


Large scale uniform poly(*o*-phenylenediamine) nanobelts can be rapidly yielded from an *o*-phenylenediamine–HAuCl<sub>4</sub> aqueous solution without the additional introduction of other templates or surfactants at room temperature.

1184

### Direct synthesis of hydrogen peroxide solution with palladium-loaded sulfonic acid polystyrene resins

Gema Blanco-Brieva, Encarnación Cano-Serrano, Jose M. Campos-Martin and Jose L. G. Fierro\*

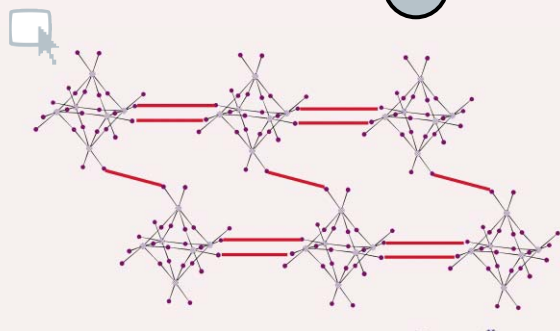


Pd loaded sulfonic acid polystyrene resins result in highly effective catalysts for the direct synthesis of H<sub>2</sub>O<sub>2</sub> using methanol as solvent; this performance is due to the ability of sulfonic acid groups to interact and stabilize Pd<sup>II</sup> ions without further reduction to Pd<sup>0</sup>.

1186

### A novel 2D net-like supramolecular polymer constructed from Ln<sub>6</sub>Cu<sub>24</sub> node and *trans*-Cu(Gly)<sub>2</sub> bridge

Jian-Jun Zhang, Sheng-Qing Xia, Tian-Lu Sheng, Sheng-Min Hu, Guido Leibel, Franc Meyer, Xin-Tao Wu,\* Sheng-Chang Xiang and Rui-Biao Fu

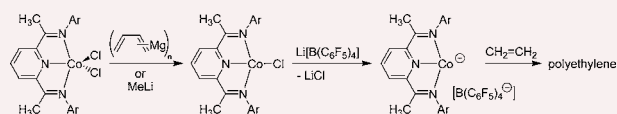


The first successful attempt to construct 2D net-like supramolecular framework with high-nuclear Ln–Cu cluster (Ln<sub>6</sub>Cu<sub>24</sub>) as node is reported.

1188

### Generation of bis(iminoalkyl)pyridine cobalt(I) cations under metal alkyl free conditions that polymerize ethene

Winfried Steffen, Tobias Blömker, Nina Kleigrewe, Gerald Kehr, Roland Fröhlich and Gerhard Erker\*

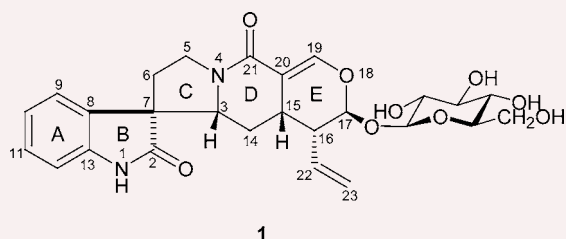


The cation complexes generated from bis(iminoalkyl)pyridine cobalt(II) dichlorides *via* the corresponding cobalt(I) chlorides show low ethene polymerization activities even under such metal alkyl free conditions.

1190

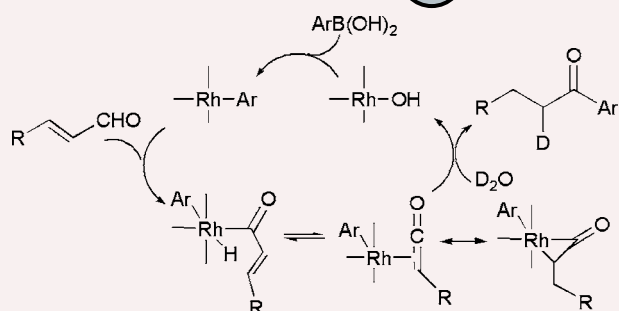
### Javaniside, a novel DNA cleavage agent from *Alangium javanicum* having an unusual oxindole skeleton

Ji Ma and Sidney M. Hecht\*



Javaniside, a novel Cu<sup>2+</sup>-dependent DNA cleavage agent, has been isolated from the leaves of the tropical tree *Alangium javanicum*.

1192

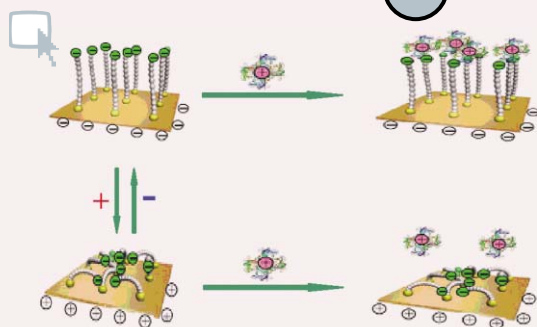


### An unprecedented rhodium-catalysed self-conjugate reduction, cross-coupling tandem reaction of cinnamaldehydes with arylboronic acids

Zhiyong Wang, Gang Zou\* and Jie Tang

A novel, unprecedented Rh-catalysed self-conjugate reduction, cross-coupling tandem reaction of cinnamaldehydes with arylboronic acids through a strained three-membered metalocyclopropanone has been disclosed and mechanistically investigated.

1194



### Controlled protein assembly on a switchable surface

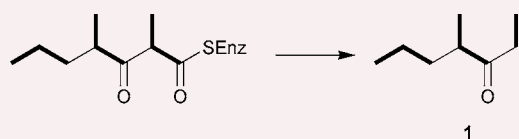
Ying Liu, Li Mu, Baohong Liu, Song Zhang, Pengyuan Yang and Jilie Kong\*

The strategy presented supplies a general method of controlling the assembly of charged proteins on loose packed SAM.

1196

### Biosynthesis of the insect pheromone (*S*)-4-methyl-3-heptanone

Andrew P. Jarvis, Jürgen Liebig, Bert Hölldobler and Neil J. Oldham\*

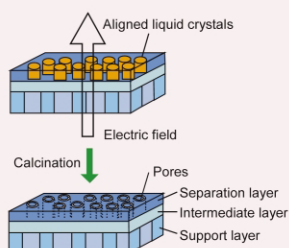


Evidence that (*S*)-4-methyl-3-heptanone and related 3-ketones are produced *de novo* from propionate and acetate units *via* a polyketide/fatty acid pathway is presented.

1198

### Preparation of a membrane with aligned nanopores using an organic–inorganic hybrid technique

Koji Kuraoka,\* Yoko Tanaka, Masaru Yamashita and Tetsuo Yazawa

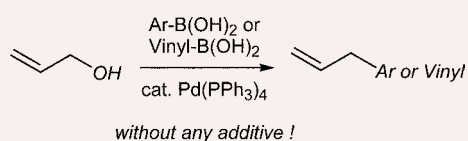


High performance molecular sieve membranes having aligned nanopores were prepared by an organic–inorganic hybrid process using an electric field to align the liquid crystal molecules therein.

1200

### Palladium(0)-catalyzed direct cross-coupling reaction of allyl alcohols with aryl- and vinyl-boronic acids

Hirokazu Tsukamoto,\* Masanori Sato and Yoshinori Kondo



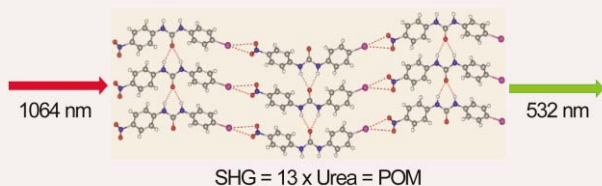
Allyl alcohols can be directly used for the palladium-catalyzed allylation of aryl- and vinyl-boronic acids without the aid of a base.

1202

### Crystal engineering of urea $\alpha$ -network via $I \cdots O_2N$ synthon and design of SHG active crystal *N*-4-iodophenyl-*N'*-4'-nitrophenylurea

Sumod George, Ashwini Nangia,\* Chi-Keung Lam,  
Thomas C. W. Mak\* and Jean-François Nicoud\*

$I \cdots O_2N$  synthon stabilizes the urea tape  $N-H \cdots O$   $\alpha$ -network in SHG active polar crystals of the title molecule.

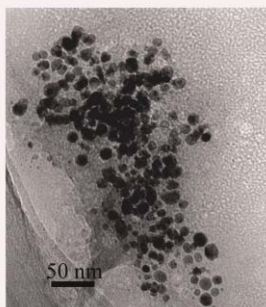


1204

### Continuous preparation of functionalised calcium phosphate nanoparticles with adjustable crystallinity

Thea Welzel, Wolfgang Meyer-Zaika and Matthias Epple\*

Both pure and functionalised calcium phosphate nanoparticles can be prepared by an easy precipitation method from aqueous solution.

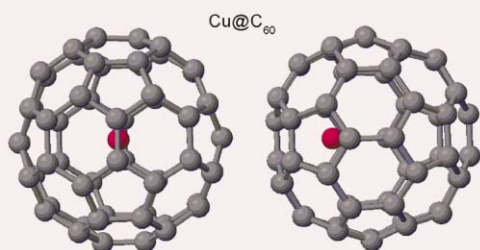


1206

### $Cu@C_{60}$ formation in rf-plasma and ring-current induced magnetism of $C_{60}$

Houjin Huang,\* Masafumi Ata and Yoko Yoshimoto

A new endofullerene,  $Cu@C_{60}$ , was synthesized and isolated. Ring-current induced diamagnetic interior and paramagnetic exterior of  $C_{60}$  have been quantitatively described using the Cu probe.

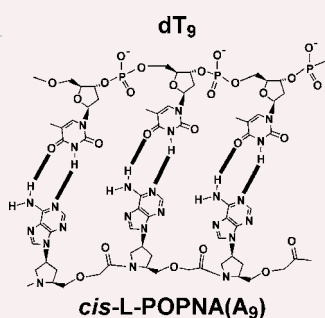


1208

### Oxy-peptide nucleic acid with a pyrrolidine ring that is configurationally optimized for hybridization with DNA

Mizuki Kitamatsu, Masanori Shigeyasu, Tomoyuki Okada and Masahiko Sisido\*

Four stereoisomers of oxy-peptide nucleic acids containing pyrrolidine rings (POPNA) were newly synthesized and investigated for binding to DNA. *cis*-L-POPNA with 9 adenine bases formed the most stable hybrid with dT<sub>9</sub>. The POPNA showed high sequence specificity and sharp melting curves in hybridization with DNA.

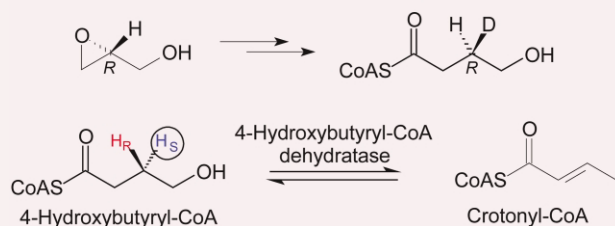


1210

### Stereochemistry of hydrogen removal from the 'unactivated' C-3 position of 4-hydroxybutyryl-CoA catalysed by 4-hydroxybutyryl-CoA dehydratase

Richard Scott, Ulrike Näser, Peter Friedrich, Thorsten Selmer,  
Wolfgang Buckel and Bernard T. Golding\*

Specific deuterium labelling has demonstrated that the pro-(*S*) hydrogen atom at C-3 of 4-hydroxybutyryl-CoA is stereospecifically abstracted by 4-hydroxybutyryl-CoA dehydratase and is not returned to C-4.

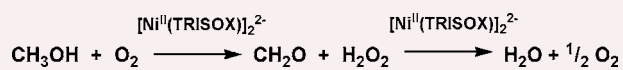


1212

**Aerobic oxidation of methanol by a Ni(II)-O<sub>2</sub> reaction**

Sara E. Edison, Richard P. Hotz and Michael J. Baldwin\*

An unusual oxygen-activating Ni(II)-oximate complex oxidizes two-hydrogen atom donating substrates, including the traditionally inert alcohol, methanol, as well as ethanol, benzyl alcohol, benzylamine, and *N*-methylbenzylamine.

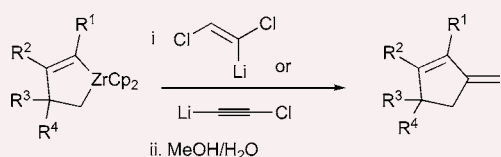


1214

**Insertion of (*E*)-(1,2-dihalovinyl)lithium and (2-haloethynyl)lithium into zirconacycles**

David Norton, Richard J. Whitby\* and Ed Griffen

Insertion of (*E*)-LiClC=CHCl or LiC≡CCl into zirconacyclopentenes affords the same methylenecyclopentenes after quenching, but by different proposed mechanisms. A zirconocene alkenylidene is a likely intermediate for the latter.

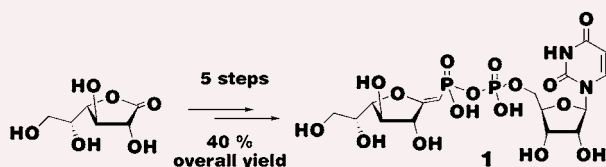


1216

**Efficient synthesis of a nucleoside-diphospho-*exo*-glycal displaying time-dependent inactivation of UDP-galactopyranose mutase**

Audrey Caravano, Stéphane P. Vincent\* and Pierre Sinaÿ\*

A short and efficient synthesis of UDP-*exo*-galactofuranosyl-glycal is presented.

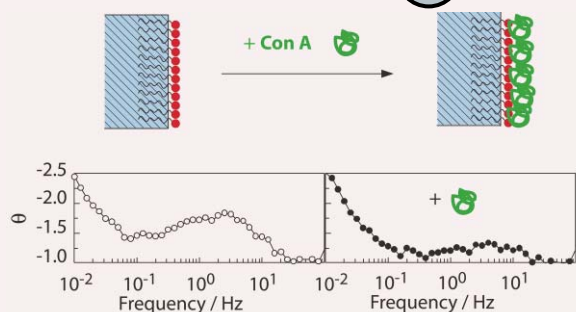


1218

**Recognition of concanavalin A at the interface between a solvent polymeric membrane and an aqueous sample monitored by electric impedance spectroscopy**

Liya Muslinkina and Ernő Pretsch\*

Electric impedance spectroscopy is used to monitor the interaction between a sugar-complexing protein and stearyl-β-D-glucopyranoside immobilized at the liquid/liquid interface between a solvent polymeric membrane and an aqueous sample.

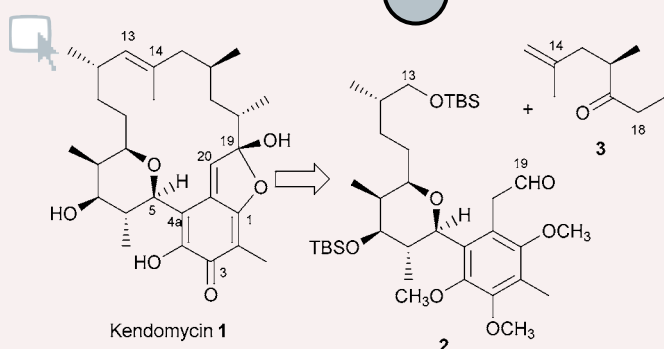


1220

**Synthetic approach to kendomycin: preparation of the C-glycosidic core**

Tetsuya Sengoku, Hirokazu Arimoto\* and Daisuke Uemura

Synthesis of the C13–C19 C-glycoside portion of kendomycin was achieved *via* oxidative pyran cyclization and Claisen rearrangement to construct the fully functionalized aromatic ring.

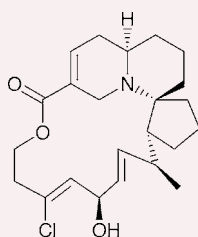




1222

**Synthesis of the tricyclic core of halichlorine**

Ichiro Hayakawa, Hirokazu Arimoto\* and Daisuke Uemura



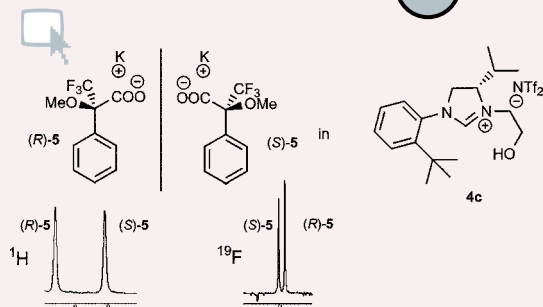
Halichlorine

Synthesis of the tricyclic core of halichlorine, a VCAM-1 expression suppressor, was achieved. The synthesis features ene-yne metathesis cyclization and regioselective oxidative cleavage of the conjugated diene.

1224

**Design and synthesis of imidazolium salts derived from (L)-valine. Investigation of their potential in chiral molecular recognition**

Hervé Clavier, Loïse Boulanger, Nicolas Audic, Loïc Toupet, Marc Mauduit\* and Jean-Claude Guillemin\*

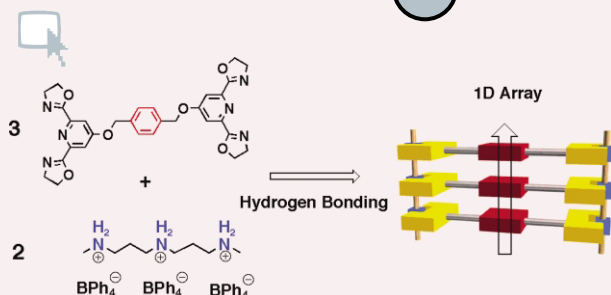


High splitting (up to 60 Hz) of the (*rac*)-potassium Mosher's salt are observed by  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectroscopy, resulting of high diastereomeric interactions with new chiral imidazolium salts derived from (L)-valine.

1226

**A one-dimensional array with controlled length from a PYBOX dimer with flexible oligo(*sec*-dialkylammonium cations)**

Takahiro Sugimoto, Kazuki Sada,\* Shigeru Sakamoto, Kentaro Yamaguchi and Seiji Shinkai

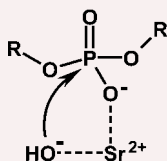


Hydrogen bonded supramolecular ladders were constructed by complexation of PYBOX dimers with flexible oligo(*sec*-dialkylammonium cations).

1228

**Phosphodiesterolytic activity of alkaline-earth cations in aqueous DMSO**

Olga Taran and Anatoly K. Yatsimirsky\*

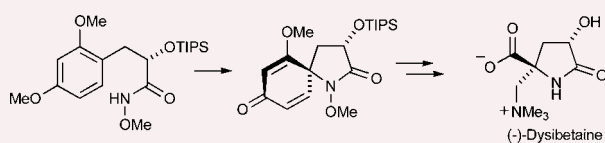


Phosphodiesterolytic activity of alkaline-earth cations strongly increases in 90% v/v aqueous DMSO in comparison with that in water.

1230

**Total synthesis of (–)-dysibetaine via a nitrenium ion cyclization–dienone cleavage strategy**

Duncan J. Wardrop\* and Matthew S. Burge



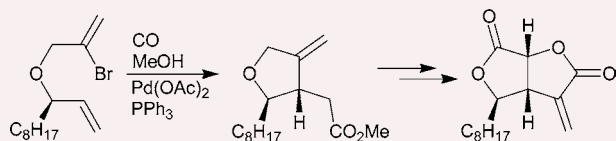
The diastereoselective total synthesis of the marine natural product (–)-dysibetaine is reported. The key steps in this venture are i) a diastereoselective nitrenium ion spirocyclization, which serves to generate the pyrrolidinone ring and quaternary stereocenter of the target, and ii) use of the 2-methoxycyclohexa-2,5-dienone ring formed during cyclization as a masked 2-amino-1,3-dicarbonyl synthon.

1232



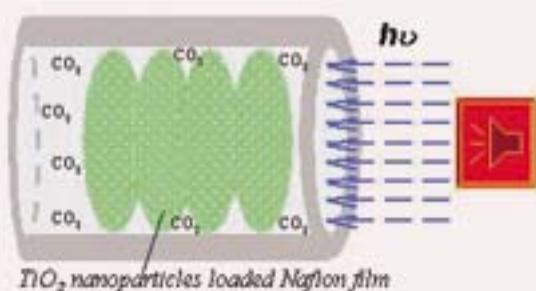
### Asymmetric synthesis of avenaciolide *via* cascade palladium catalysed cyclisation–carbonylation of bromodienes

Varinder K. Aggarwal,\* Paul W. Davies and Andreas T. Schmidt



An asymmetric synthesis of the secondary metabolite avenaciolide has been achieved utilising a diastereoselective palladium catalysed cyclisation–carbonylation of bromodienes.

1234



### Improving photoreduction of CO<sub>2</sub> with homogeneously dispersed nanoscale TiO<sub>2</sub> catalysts

Pankaj Pathak, Mohammed J. Meziani, Yin Li, LaShonda T. Cureton and Ya-Ping Sun\*

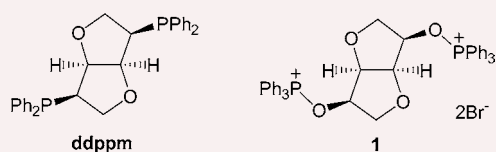
Significantly improved photoconversion of CO<sub>2</sub> was achieved with photocatalysts of nanoscale TiO<sub>2</sub> particles homogeneously dispersed in porous cavities of optically transparent ionomer membrane thin films.

1236



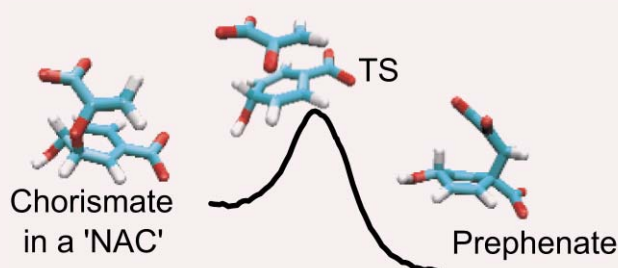
### A concise synthesis of a rigid isomannide-based diphosphine ligand and structural characterisation of an alkoxyphosphonium intermediate

Cristina Carcedo, Athanasia Dervisi,\* Ian A. Fallis, Liling Ooi and K. M. Abdul Malik



The synthesis of the rigid chelating diphosphine ligand **ddppm** and its applications in asymmetric hydrogenations are discussed. The intermediate secondary bis-alkoxyphosphonium salt (**1**) was isolated in high yield and structurally characterised.

1238



### Conformational effects in enzyme catalysis: QM/MM free energy calculation of the 'NAC' contribution in chorismate mutase

Kara E. Ranaghan and Adrian J. Mulholland\*

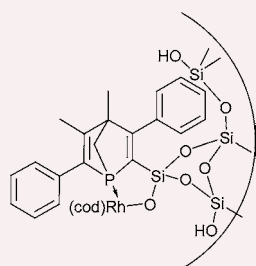
The 'near attack conformation' (NAC) effect in chorismate mutase is calculated to contribute 3.8–4.6 kcal mol<sup>-1</sup>, indicating that the NAC effect by itself does not account for catalysis in this enzyme.

1240



### New P<sup>+</sup>O ligand grafted on periodically organised mesoporous silicas for one-pot bifunctional catalysis: Coupling of base catalysed Knoevenagel condensation with *in situ* Rh catalysed hydrogenation

Frédéric Goettmann, David Grosso, François Mercier, François Mathey and Clément Sanchez\*



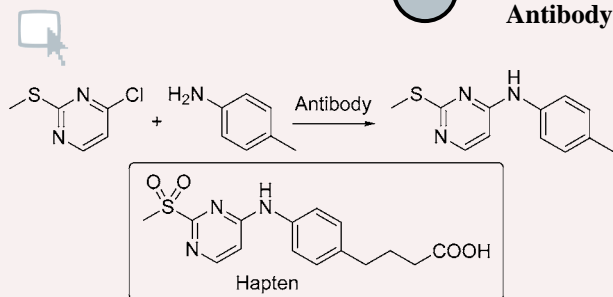
New phosphanorbornadiene rhodium hydride mesostructured silicas behave as very active recyclable hydrogenation catalysts which combined with additional grafted amino functions allow efficiently driven one-pot bifunctional catalysis.

1242

**Antibody-catalyzed aminolysis of a chloropyrimidine derivative**

Fujie Tanaka,\* Fabiola Flores, Diane Kubitz, Richard A. Lerner\* and Carlos F. Barbas, III\*

Aminolysis of a chloropyrimidine derivative was faster in water than in MeOH or CH<sub>3</sub>CN. Antibody FTB8E9 was generated by immunization with the designed hapten to catalyze the reaction.

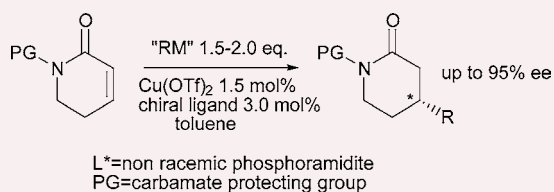


1244

**Unprecedented copper-catalyzed asymmetric conjugate addition of organometallic reagents to  $\alpha,\beta$ -unsaturated lactams**

Mauro Pineschi,\* Federica Del Moro, Francesca Gini, Adriaan J. Minnaard and Ben L. Feringa

The first enantioselective conjugate addition of hard organometallic reagents (alkylzincs and alkylaluminiums) to  $\alpha,\beta$ -unsaturated lactams bearing appropriate protecting-activating groups on the nitrogen atom is described.



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