**Cover (far left)**

The molecular structure design approach to construction of heterometallic alkoxide complexes can be compared to LEGO® building with stable coordination polyhedra. Photo courtesy of Camille Goudeseune (<http://zx81.isl.uiuc.edu/lego/diaspar/>) (pp. 1213–1222).

**Inside cover (left)**

Phase transition between crystalline and liquid-crystalline phases (and vice-versa) of Eu(III) containing mesogens are detected by monitoring luminescence intensity and lifetime (pp. 1226–1227).

# contents

## FOCUS ARTICLE

1209

? ionic liquids \$

? ionic liquids \$

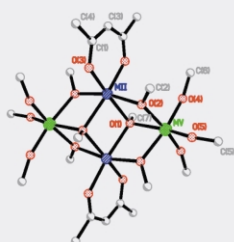
**From curiosities to commodities: ionic liquids begin the transition**

James H. Davis, Jr. and Phillip A. Fox

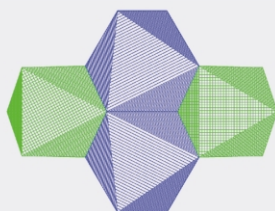
No longer regarded as chemical curiosities, ionic liquids are making inroads in research programs the world over. This trend is being facilitated by the rapidly growing number of ionic liquids available from commercial suppliers, who are now making ionic liquids available on industrial scales.

## FEATURE ARTICLE

1213



=

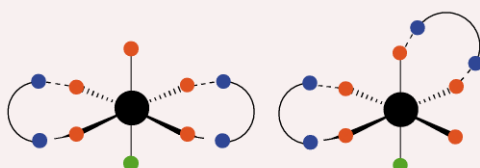
**Molecular structure design and synthetic approaches to the heterometallic alkoxide complexes (soft chemistry approach to inorganic materials by the eyes of a crystallographer)**

Vadim G. Kessler

General principles of formation and stability of the heterometallic alkoxides existing due to Lewis Acid–Base interaction, isomorphous substitution and heterometallic metal–metal bonds are discussed in connection with the molecular structure design approach.

## COMMUNICATIONS

1224

**Charge assisted chiral hybrid H-bonded molecular networks**

Sylvie Ferlay, Roman Holakovský, Mir Wais Hosseini,\* Jean-Marc Planeix and Nathalie Kyritsakas

Self-assembly of chiral dicationic tectons and  $[\text{Fe}(\text{CN})_5\text{NO}]^{2-}$  anion leads to the formation of infinite neutral chiral H-bonded molecular networks in the crystalline phase.

Chemical Communications  
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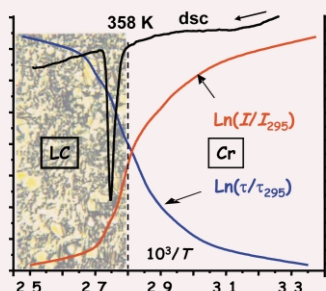
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1226

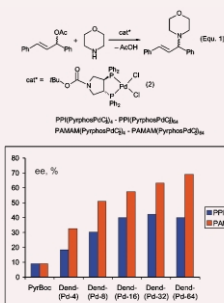


### Luminescence-detected phase transitions in lanthanide-containing liquid crystals

Stéphane Suárez, Olimpia Mamula, Daniel Imbert, Claude Piguet and Jean-Claude G. Bünzli\*

Variations of luminescence intensity and lifetime *versus* temperature reflect phase transitions occurring in a europium-containing macrocyclic complex with metallomesogen properties.

1228

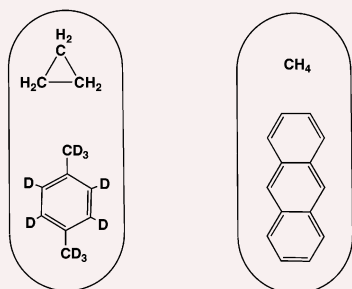


### A strongly positive dendrimer effect in asymmetric catalysis: allylic aminations with Pyrphos-palladium functionalised PPI and PAMAM dendrimers

Yann Ribourdouille, Gerald D. Engel, Mireille Richard-Plouet and Lutz H. Gade\*

The first example of a strongly positive (enantio)selectivity effect in dendrimer catalysis is reported for the asymmetric allylic aminations catalysed by high-generation Pyrphos-palladium functionalised PPI and PAMAM dendrimers.

1230

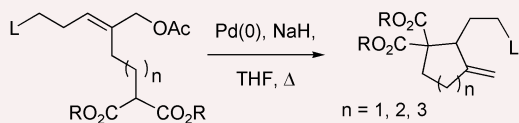


### Coencapsulation of large and small hydrocarbons

Alexander Shivanyuk, Alessandro Scarso and Julius Rebek, Jr.\*

Combinations of small, gaseous hydrocarbons and sizable aromatics, *e.g.* methane and anthracene, are directly observed in a cylindrical capsule by NMR.

1232

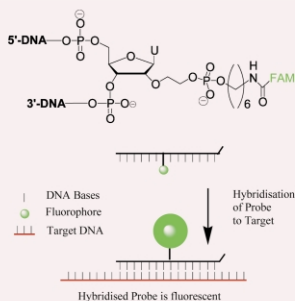


### Palladium-catalyzed, heteroatom assisted, regioselective cyclizations

Marie E. Krafft\* and Matthew C. Lucas

Unusual regioselective Pd-catalyzed cyclizations of unsymmetrically substituted allylic acetates demonstrate the remarkable power of directing groups in imparting significant control over carbocyclizations *via* preassociation.

1234

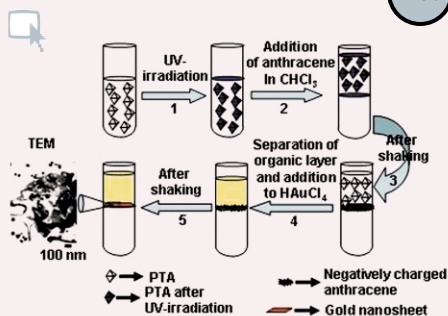


### Synthesis of HyBeacons and dual-labelled probes containing 2'-fluorescent groups for use in genetic analysis

Neil Dobson, David G. McDowell, David J. French, Lynda J. Brown, John M. Mellor and Tom Brown\*

A protected 2'-hydroxyethyl uridine phosphoramidite has been used to synthesise fluorescein-labelled HyBeacon probes and dual-labelled fluorogenic oligonucleotides.

1236

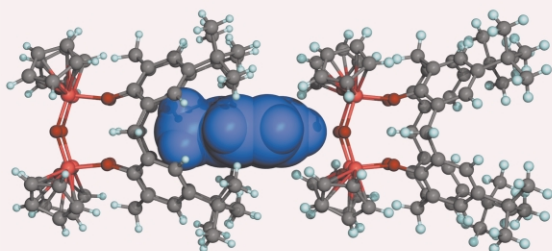


### Gold nanosheets *via* reduction of aqueous chloroaurate ions by anthracene anions bound to a liquid–liquid interface

Ambarish Sanyal and Murali Sastry\*

Scheme showing the different steps leading to the formation of gold nanosheets at the liquid–liquid interface by reduction of chloroaurate ions using negatively charged anthracene.

1238

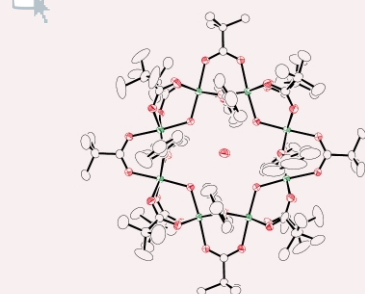


### Titanocene dichloride route to a tetranuclear oxo-bridged monocyclopentadienyl titanium(IV) calix[4]arene complex

Antonella J. Petrella, Nicholas K. Roberts, Colin L. Raston, Mark Thornton-Pett and Robert N. Lamb

*In situ* lithiation of *p*-<sup>t</sup>Bu-calix[4]arene then reaction with titanocene dichloride affords a tetranuclear titanium monocyclopentadienyl complex with each O-phenolate moiety bound to CpTi(IV), and with the metal centres μ-oxo bridged, in a  $-(TiO)_4$  ring system.

1240

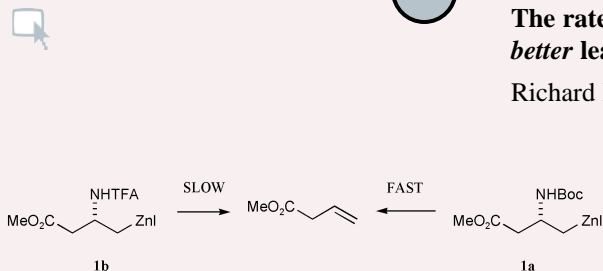


### A phenolysis route to a new type of octanuclear iron(III) wheel: $[Fe_8(OH)_4(OPh)_8(O_2CBu^t)_{12}]$

Cristina Cañada-Vilalta, Maren Pink and George Christou\*

The reaction of a hexanuclear iron(III) compound with different alcohols has afforded two new ferric wheels: methanol yields  $[Fe_{10}(OMe)_{20}(O_2CBu^t)_{10}]$ , whereas phenol gives  $[Fe_8(OH)_4(OPh)_8(O_2CBu^t)_{12}]$  with an unprecedented type of wheel structure for Fe.

1242

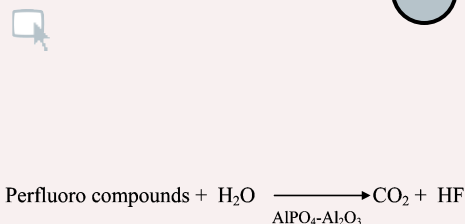


### The rate of elimination of a β-amino zinc reagent is *reduced* by using a *better* leaving group

Richard F. W. Jackson,\* Ian Rilatt and P. John Murray

The trifluoroacetyl-protected β-amino organozinc reagent **1b** is, surprisingly, more stable towards elimination than the corresponding Boc-protected analogue **1a**.

1244



### Hydrolytic decomposition of PFCs over $AlPO_4-Al_2O_3$ catalyst

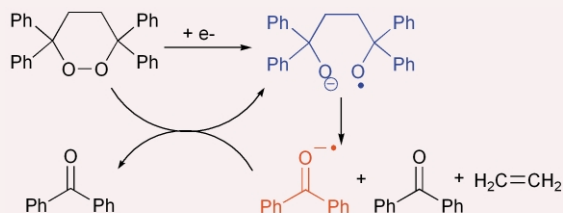
Jong Yeol Jeon, Xiu-Feng Xu, Mi Hwa Choi, Hee Young Kim and Yong-Ki Park\*

The conveniently and cheaply prepared catalyst  $AlPO_4-Al_2O_3$  catalyst is used to effectively decompose  $CF_4$  into  $CO_2$  and HF without formation of any by-products and without catalyst deactivation.

1246

**Radical anion chain process initiated by a dissociative electron transfer to a monocyclic endoperoxide**

Donald L. B. Stringle, R. Nevin Campbell and Mark S. Workentin\*

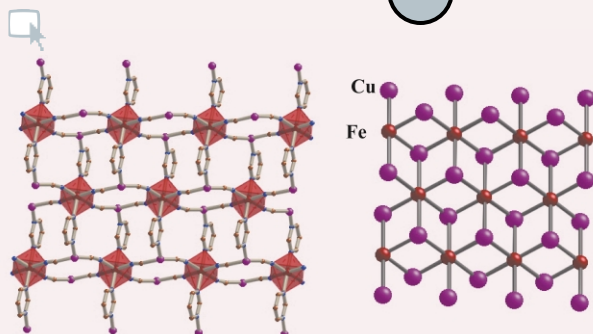


Electron transfer to 3,3,6,6-tetraphenyl-1,2-dioxane results in the cleavage of the O–O bond, generating a distonic radical anion that fragments and initiates an unprecedented radical anion chain process.

1248

**Cooperative thermal and optical switching of spin states in a new two-dimensional coordination polymer**

Virginie Niel, Ana Galet, Ana B. Gaspar, M. Carmen Muñoz and José A. Real\*

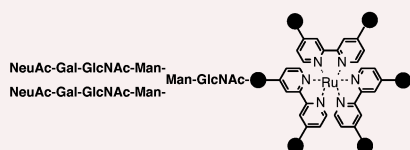


{Fe(pyrimidine)<sub>2</sub>[Cu(CN)<sub>2</sub>]<sub>2</sub>} a switchable two-dimensional spin crossover coordination polymer with CdCl<sub>2</sub> network topology.

1250

**Ruthenium complexes carrying a disialo complex-type oligosaccharide: enzymatic synthesis and its application to a luminescent probe to detect influenza viruses**

Shinji Kojima, Teruaki Hasegawa, Takahiro Yonemura, Ken Sasaki, Kenji Yamamoto, Yutaka Makimura, Tadanobu Takahashi, Takashi Suzuki, Yasuo Suzuki and Kazukiyo Kobayashi\*

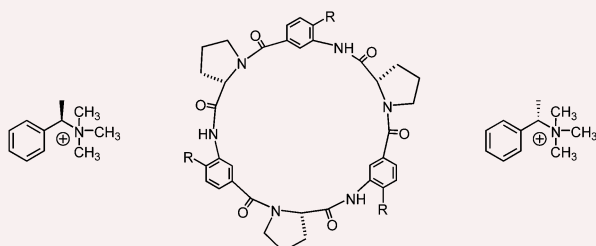


Tris-bipyridine ruthenium-complexes carrying a disialo complex-type oligosaccharide are reported which bind to type-A influenza viruses with excellent affinity ( $IC_{50} = 8.4 \mu M$ ).

1252

**Enantioselective recognition of a chiral quaternary ammonium ion by C<sub>3</sub> symmetric cyclic hexapeptides**

Guido Heinrichs, Laurent Vial, Jérôme Lacour and Stefan Kubik\*

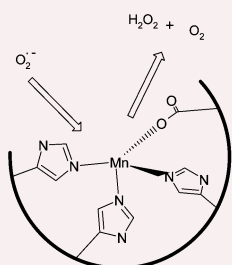


Cyclic hexapeptides containing aromatic amino acid subunits represent the first class of artificial cation hosts with which a significant level of enantioselective recognition of chiral quaternary ammonium ions can be achieved.

1254

**A polymer supported manganese catalyst useful as a superoxide dismutase mimic**

Theeraphon Piacham, Chartchalerm Isarankura Na Ayudhya, Virapong Prachayasittikul, Leif Bülow and Lei Ye\*



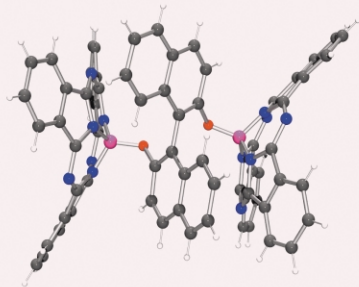
Polymer supported manganese was synthesized *via* a template polymerization involving functional monomers to afford a catalyst with superoxide dismutase activity.

1256

**An optically-active subphthalocyanine dimer**

Takamitsu Fukuda, Marilyn M. Olmstead, William S. Durfee\* and Nagao Kobayashi\*

A subphthalocyanine (SubPc) dimer linked by optically-active (*R*)-(+)-BINOL has been made and studied using CD and MCD spectroscopies. The X-ray crystal structure of the racemic dimer has been determined.

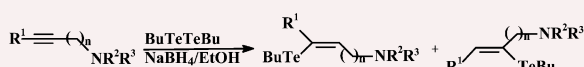


1258

**Hydrotelluration of aminoalkynes**

Gilson Zeni,\* Olga Soares do Rego Barros, Angélica Venturini Moro, Antonio Luiz Braga and Clovis Peppe

The first hydrotelluration reaction of aminoalkynes by metal tellurolates. Both the nature of the metallic counter ion and the degree of substitution on the amino substituent play important roles in the regio-, and stereo-chemistry of the reaction.

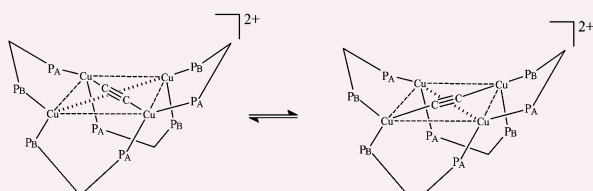


1260

**An oscillating C<sub>2</sub><sup>2-</sup> unit inside a copper rectangle**

Wing-Yin Lo, Chi-Ho Lam, Wendy Kit-Mai Fung, Hong-Zhe Sun, Vivian Wing-Wah Yam,\* David Balcells, Feliu Maseras and Odile Eisenstein\*

The fluxional behaviour of [Cu<sub>4</sub>(μ-dppm)<sub>4</sub>(μ<sub>4</sub>-η<sup>1</sup>,η<sup>2</sup>-C≡C-)]<sup>2+</sup> has been studied by <sup>31</sup>P and <sup>1</sup>H NMR spectroscopy, and has been supported by DFT(B3LYP) calculations.

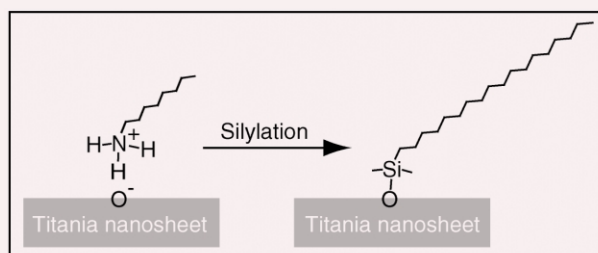


1262

**Surface modification of a layered alkali titanate with organosilanes**

Yusuke Ide and Makoto Ogawa\*

Silylated derivatives of K<sub>2</sub>Ti<sub>4</sub>O<sub>9</sub>·*n*H<sub>2</sub>O were successfully synthesized by the reaction of the octylammonium–tetratitanate intercalation compound with *n*-alkyltrimethoxysilanes. The silylated derivatives showed improved thermal and chemical stabilities compared with the corresponding octylammonium derivative, and swell in organic solvents.

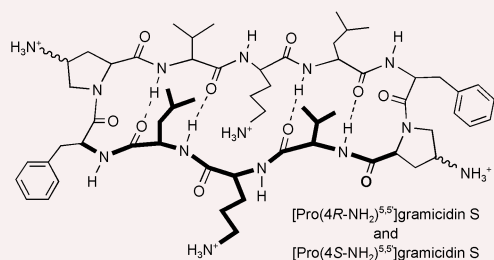


1264

**Extra amino group-containing gramicidin S analogs possessing outer membrane-permeabilizing activity**

Masao Kawai,\* Ryoji Tanaka, Hatsuo Yamamura, Keiko Yasuda, Shizuto Narita, Hiroshi Umemoto, Setsuko Ando and Takashi Katsu

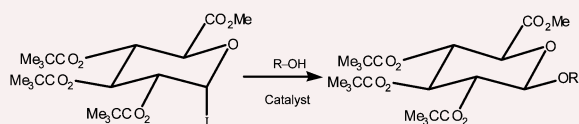
The unique analogs of the cyclic decapeptide antibiotic gramicidin S containing (2*S*,4*R*/*S*)-4-aminoproline residues were synthesized. The tetracationic analogs exhibited marked permeabilizing activity on the outer membrane of gram-negative bacteria.



1266

**Preparation, X-ray structure and reactivity of a stable glycosyl iodide**

Jamie Bickley, Jennifer A. Cottrell, John R. Ferguson, Robert A. Field, John R. Harding, David L. Hughes, K. P. Ravindanathan Kartha, Jayne L. Law, Feodor Scheinmann and Andrew V. Stachulski\*

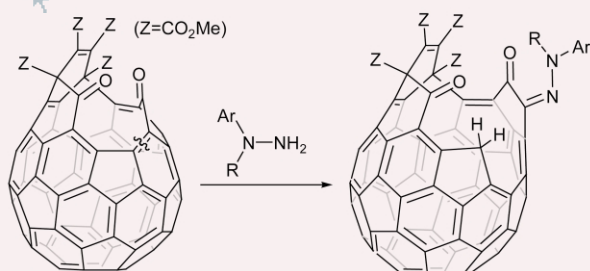


The preparation, stability, X-ray crystal structure and glycosyl donor reactivity of a 'disarmed' glycosyl iodide are reported.

1268

**A novel migrative addition reaction of hydrazines to the diketone derivative of C<sub>60</sub>**

Sho-ichi Iwamatsu,\* Fumiaki Ono and Shizuaki Murata

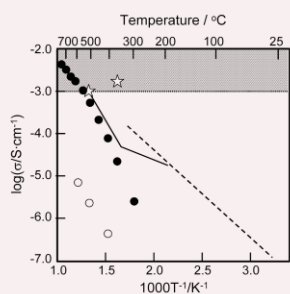


An addition reaction of an aromatic hydrazine to the diketone derivative of C<sub>60</sub> occurs highly regioselectively with an unusual migration to give a fluorescent product having a methylene carbon along the orifice.

1270

**A new type of bromide anion conducting solid**

Nobuhito Imanaka\* and Yasuhiro Kato

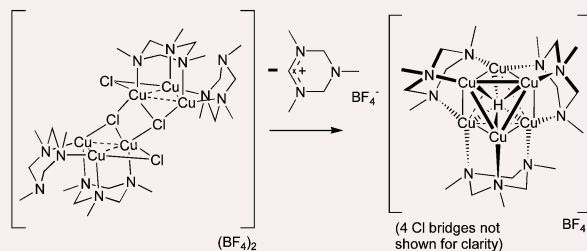


We report on a La<sub>0.9</sub>Sr<sub>0.1</sub>OBr<sub>0.9</sub> solid electrolyte with a high Br<sup>-</sup> anion conductivity of  $4.2 \times 10^{-3} \text{ S cm}^{-1}$  at 750 °C, which lies in the superionic conductor region ( $>10^{-3} \text{ S cm}^{-1}$ , shaded area). The highest Br<sup>-</sup> anion conductivities for conventional CsPbBr<sub>3</sub> and PbBr<sub>2</sub> solids are still below this region due to their low melting points.

1272

**Trimethyltriazacyclohexane as bridging ligand for triangular Cu<sub>3</sub> units and C–H hydride abstraction into a Cu<sub>6</sub> cluster**

Randolf D. Köhn,\* Zhida Pan, Mary F. Mahon and Gabriele Kociok-Köhn

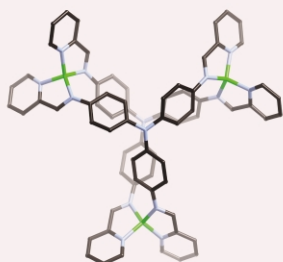


A triazacyclohexane without pendant arm donor groups can be coordinated to a triangular Cu<sub>3</sub> cluster and leads to hydride abstraction into an octahedral Cu<sub>6</sub> cluster.

1274

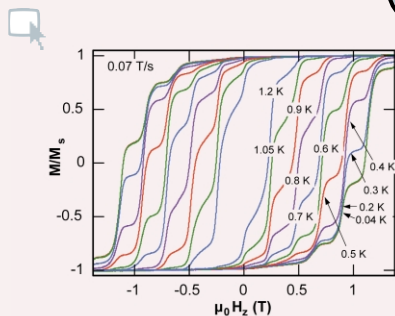
**The 'Trinity' helix: synthesis and structural characterisation of a C<sub>3</sub>-symmetric tris-bidentate ligand and its coordination to Ag(I)**

Brian Conerney, Paul Jensen, Paul E. Kruger\* and Conchúir MacGloinn



A rigid trigonal ligand **L** is shown to readily coordinate Ag(I) to form a trinuclear double helicate [Ag<sub>3</sub>L<sub>2</sub>]<sup>3+</sup> species as characterised by <sup>1</sup>H NMR compleximetric titration and ESMS. Employment of a C<sub>3</sub>-symmetric ligand represents a significant move away from the traditionally employed C<sub>2</sub>-symmetric ligand types.

1276

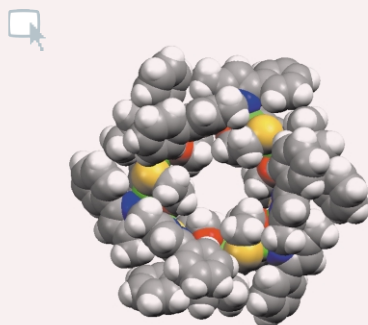


### Dodecanuclear and octanuclear manganese rods

Euan K. Brechin,\* Monica Soler, George Christou, Madeleine Helliwell, Simon J. Teat and Wolfgang Wernsdorfer

Dodecanuclear and octanuclear manganese clusters formed from edge-sharing triangular units give unusual rod-like structures with high spin ground states and single-molecule magnetism behaviour.

1278

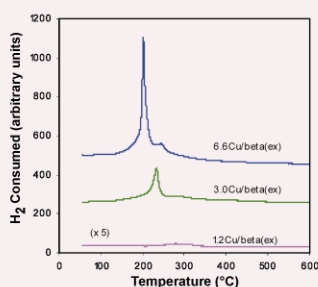


### Synthesis and structure of the hexameric, dodecanuclear metallamacrocycle [(5-methyl-3-phenylpyrazole)<sub>2</sub>Zn<sub>2</sub>(OCH<sub>2</sub>CH<sub>2</sub>S)]<sub>6</sub>

David T. Puerta and Seth M. Cohen\*

The largest member of a family of metallamacrocycles that utilize bridging pyrazole ligands has been synthesized and structurally characterized in the solid state.

1280

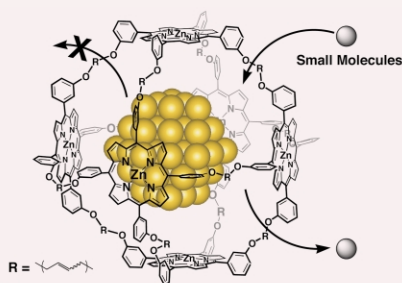


### Copper exchanged beta zeolites for the catalytic oxidation of ammonia

Teresa Curtin\* and Sandra Lenihan

Copper exchanged on beta zeolites are extremely active and selective for the catalytic oxidation of ammonia to nitrogen and water and this activity correlates to the ease of reduction of the copper species.

1282

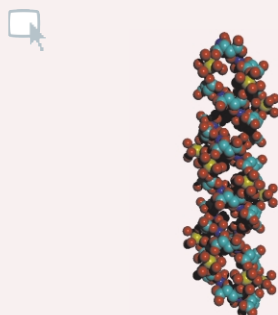


### Gold nanocluster confined within a cage: template-directed formation of a hexaporphyrin cage and its confinement capability

Tomohiko Inomata and Katsuaki Konishi\*

'Caged' gold cluster was synthesized by cross-linking among zinc porphyrins assembled around a cluster; the cage showed notable confinement capability for the cluster core, but allowed the access of small molecules to the cluster surface.

1284



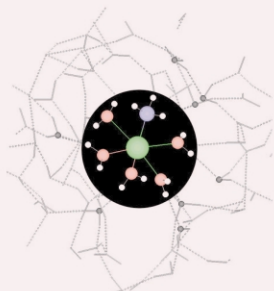
### A novel left-handed double helicate constructed from L-tartrate bridged molybdenum(VI) and gadolinium(III) atoms

Chuan-De Wu, Can-Zhong Lu,\* Xiang Lin, Ding-Ming Wu, Shao-Fang Lu, Hong-Hui Zhuang and Jin-Shun Huang

The one-dimensional double helicate,  $[\text{NH}_4][\text{Mo}_2\text{O}_4\text{Gd}(\text{H}_2\text{O})_6(\text{L-C}_4\text{H}_2\text{O}_6)_2] \cdot 4\text{H}_2\text{O}$ , is built up by two left-handed single-helical chains, linked up further by eight-coordinated  $\text{Gd}^{\text{III}}$  pieces in an enantiopure left-handed double helical configuration, of which each helix is formed by L-tartrate bridged six-coordinated  $\text{Mo}^{\text{VI}}$  atoms.



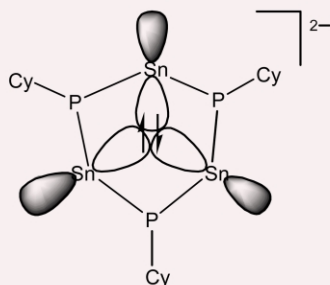
1286

**The influence of the Jahn–Teller effect and of heteroligands on the reactivity of Cu<sup>2+</sup>**

Christian F. Schwenk and Bernd M. Rode\*

Quantum-mechanics-based simulations have supplied experimentally hardly accessible data for the Cu<sup>2+</sup> ion and its amino complexes in water, as the dynamic Jahn–Teller effect and ligand exchange processes in the picosecond time scale. New insights into the process of the Jahn–Teller inversions and the biochemically relevant influence of heteroligands on the reactivity of Cu<sup>2+</sup> have been obtained.

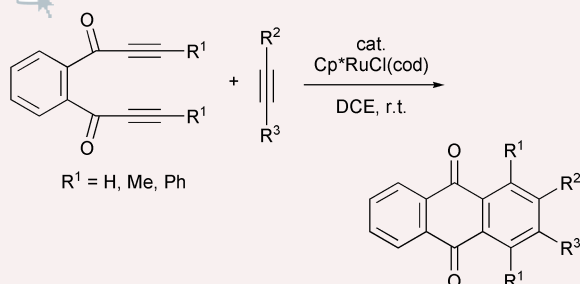
1288

**Synthesis and structure of [{Sn(μ-PCy)}<sub>3</sub>(Na·PMDETA)<sub>2</sub>], containing an electron-deficient [{Sn(μ-PCy)}<sub>3</sub>]<sup>2-</sup> dianion**

Paula Alvarez-Bercedo, Andrew D. Bond, Robert Haigh, Alexander D. Hopkins, Gavin T. Lawson, Mary McPartlin, David Moncrieff, Marta E. Gonzalez Mosquera, Jeremy M. Rawson, Anthony D. Woods and Dominic S. Wright\*

The title compound [{Sn(μ-PCy)}<sub>3</sub>(Na·PMDETA)<sub>2</sub>] (**1**), contains an electron-deficient [{Sn(μ-PCy)}<sub>3</sub>]<sup>2-</sup> dianion with a novel 2-electron 3-centre (2e–3c) bonding arrangement.

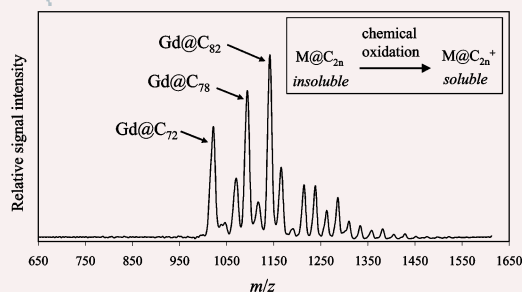
1290

**Ru(II)-catalyzed [2 + 2 + 2] cycloaddition of 1,2-bis(propioyl)benzenes with monoalkynes leading to substituted anthraquinones**

Yoshihiko Yamamoto,\* Koichi Hata, Takayasu Arakawa and Kenji Itoh

[2 + 2 + 2] cycloadditions of 1,2-bis(propioyl)benzenes with monoalkynes are effectively catalysed by Cp<sup>\*</sup>RuCl(cod) under mild conditions to give substituted anthraquinones in moderate to high yields.

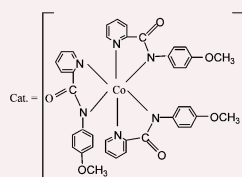
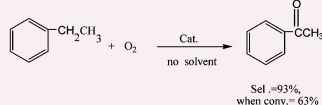
1292

**Chemical oxidation of endohedral metallofullerenes: identification and separation of distinct classes**

Robert D. Bolskar\* and J. Michael Alford

Chemical oxidation is used to solubilize and separate previously unused fractions of metallofullerenes, including three fractions of Gd@C<sub>2n</sub> species, and has allowed the solubilization of Tm@C<sub>60</sub><sup>+</sup> and Tm@C<sub>70</sub><sup>+</sup> species for the first time.

1294

**Synthesis and characterization of cobalt(III) complexes containing 2-pyridinecarboxamide ligands and their application in catalytic oxidation of ethylbenzene with dioxygen**

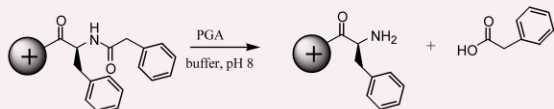
Jian-Ying Qi, Hong-Xia Ma, Xian-Jun Li,\* Zhong-Yuan Zhou, Michael C. K. Choi, Albert S. C. Chan\* and Qi-Yun Yang

Four novel cobalt(III) complexes were found to have high catalytic activities and excellent selectivities in the oxidation of ethylbenzene to acetophenone using O<sub>2</sub> as oxidant without need of solvent.

1296

**Improved biotransformations on charged PEGA supports**

Alessandra Basso, Luigi De Martin, Lucia Gardossi,\* Graham Margetts, Ian Brazendale, Annie Y. Bosma, Rein V. Ulijn\* and Sabine L. Flitsch



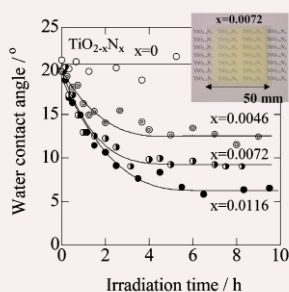
A novel positively charged PEGA resin significantly improves penicillin G amidase (PGA) catalysed biotransformation on solid support, by favouring accessibility of the negatively charged enzyme.

1298

**Visible-light induced hydrophilicity on nitrogen-substituted titanium dioxide films**

Hiroshi Irie, Seitaro Washizuka, Norio Yoshino and Kazuhito Hashimoto\*

We have fabricated  $\text{TiO}_{2-x}\text{N}_x$  thin films which undergo hydrophilic conversion under visible light irradiation. The hydrophilicity was enhanced by increasing the degree of nitrogen substitution at oxygen sites.

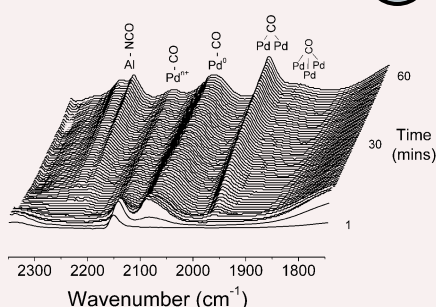


1300

**In situ ammonia generation as a strategy for catalytic NO<sub>x</sub> reduction under oxygen rich conditions**

Norman Macleod and Richard M. Lambert\*

A novel low temperature mechanism for lean NO<sub>x</sub> reduction over Pd/Al<sub>2</sub>O<sub>3</sub> catalysts has been identified involving formation and subsequent hydrolysis of NCO species, generating the active reductant NH<sub>3</sub> *in situ*.

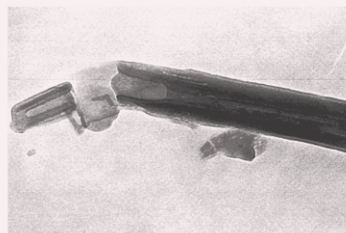


1302

**Synthesis of silica nanotubes from kaolin clay**

Wenjun Dong, Wenjiang Li, Kaifeng Yu, K. Krishna, Lizhu Song, Xiaofeng Wang, Zichen Wang,\* Marc-Olivier Coppens and Shouhua Feng\*

Silica nanotubes were synthesized from kaolin clay using surfactant intercalation, sulfuric acid and hydrothermal treatments. These tubular structures had open ends, an inner diameter of about 50 nm and an outer diameter of 80 nm, and a length of less than 1 μm on average.

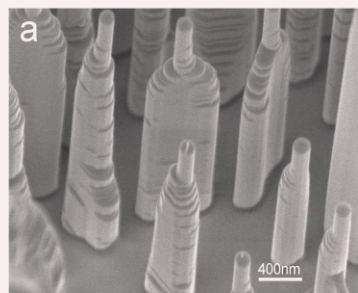


1304

**Tower-like structure of ZnO nanocolumns**

Ping'an Hu, Yunqi Liu,\* Xianbiao Wang, Lei Fu and Daoben Zhu\*

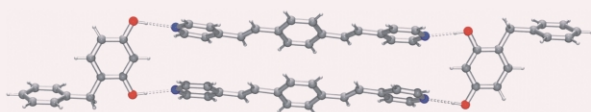
A novel nanostructure: a tower-like ZnO nanocolumn is synthesized by a catalyst-free method. The ZnO nanocolumns with layer structures are highly crystalline and normal to alumina substrates.



1306

**'Template-switching': a supramolecular strategy for the quantitative, gram-scale construction of a molecular target in the solid state**

Tomislav Friščić and Leonard R. MacGillivray\*

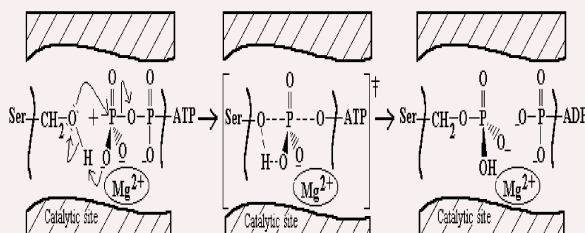


A template-switching strategy exploits the modularity of template-directed solid-state synthesis to achieve a quantitative, gram-scale construction of a *p*-[2.2]-cyclophane target in the solid state.

1308

**Density functional study of the enzymatic reaction catalyzed by a cyclin-dependent kinase**

Andrea Cavalli, Marco De Vivo and Maurizio Recanatini\*

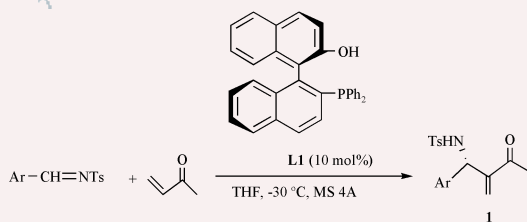


The phosphoryl transfer reaction catalyzed by CDK2 was investigated for the first time by means of quantum mechanical calculations.

1310

**Chiral phosphine Lewis base catalyzed asymmetric aza-Baylis–Hillman reaction of *N*-sulfonated imines with methyl vinyl ketone and phenyl acrylate**

Min Shi\* and Lian-Hui Chen



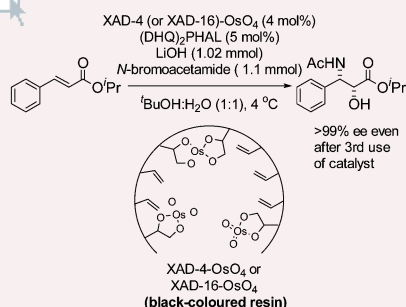
Ar could be various substituted phenyl groups, yield: 82%–96%, ee: 76–94%.

In the presence of a catalytic amount of chiral phosphine Lewis base **L1** (10 mol%) and molecular sieve 4A, the aza-Baylis–Hillman adducts **1** could be obtained in good yields with high ee.

1312

**Osmylated macroporous resins: safe, highly efficient and recyclable catalysts for asymmetric aminohydroxylation of olefins**

Cheon Hee Jo, Sien-Ho Han, Jung Woon Yang, Eun Joo Roh, Ueon-Sang Shin and Choong Eui Song\*

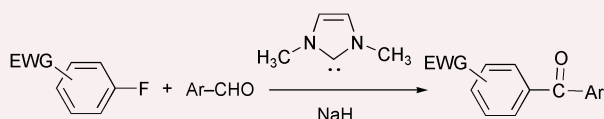


Osmylated macroporous resins showed excellent catalytic efficiencies in the asymmetric aminohydroxylation of olefins and, moreover, these resins were easily recovered and reused without any significant decrease in catalytic performances.

1314

**Nucleophilic acylation of arylfluorides catalyzed by imidazolidenyl carbene**

Yumiko Suzuki,\* Tomonori Toyota, Fumie Imada, Masayuki Sato and Akira Miyashita



Catalytic action of *N*-heterocyclic carbene. Synthesis of benzophenones from arylfluorides and benzaldehydes by imidazolidenyl carbene-catalyzed nucleophilic acylation.

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