

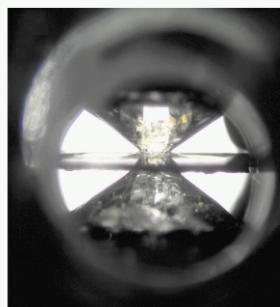
Cover (far left)
TEM image of monodisperse 11 nm γ -Fe₂O₃ nanoparticles. Photo taken by G. Markovich and P. Poddar (pp. 927-932).

Inside cover (left)
The structure of a single-molecule capsule sealed by recognition of the ion-pair Me₆N⁺ (gold) and Cl⁻ (green). Image generated using the program Xseed (<http://www.x-seed.net>) (pp. 940-941).

contents

FOCUS ARTICLE

919



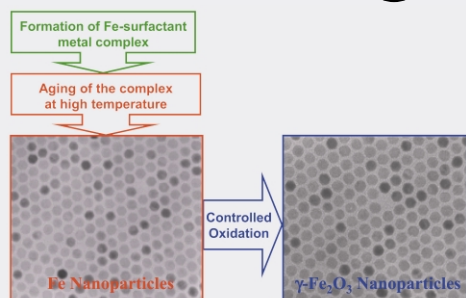
Chemistry of materials under extreme high pressure-high-temperature conditions

Paul F. McMillan

The author describes how the development of diamond anvil cells is changing the study of solid state and liquid phase chemistry under extreme high pressure-high temperature conditions.

FEATURE ARTICLE

927



Chemical synthesis of magnetic nanoparticles

Taeghwan Hyeon

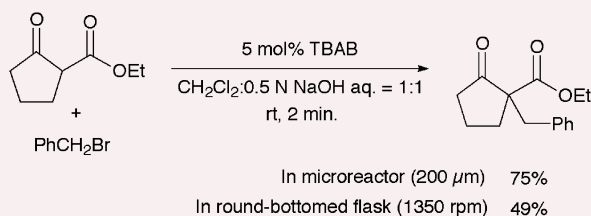
This Feature Article reviews recent advances in the synthesis of various magnetic nanoparticles using colloidal chemical approaches.

COMMUNICATIONS

936

Phase-transfer alkylation reactions using microreactors

Masaharu Ueno, Hideaki Hisamoto, Takehiko Kitamori and Shū Kobayashi*



Phase-transfer alkylation in a microreactor proceeds smoothly, and the reaction has been found to be more efficient than that in a round-bottomed flask with vigorous stirring.

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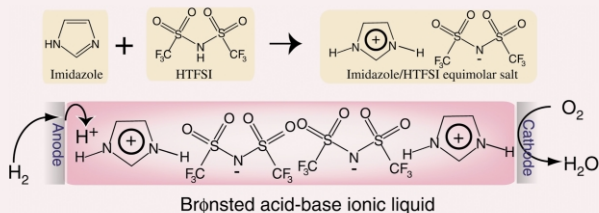
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Brønsted acid–base ionic liquids and their use as new materials for anhydrous proton conductors

Md. A. B. H. Susan, Akihiro Noda, Shigenori Mitsushima and Masayoshi Watanabe*



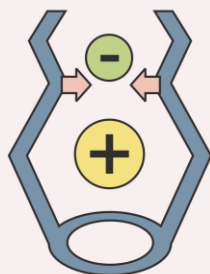
Novel Brønsted acid–base ionic liquids, are electroactive for H₂ oxidation and O₂ reduction at a Pt electrode and have the prospect of use for anhydrous proton conductors at elevated temperatures.

940

Anion-sealed single-molecule capsules

Jerry L. Atwood* and Agnieszka Szumna

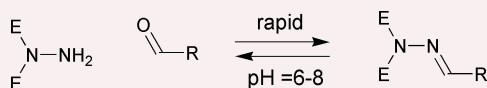
A new approach to anion recognition utilizing electrostatic and hydrogen bonding interactions has been demonstrated by placement of the whole ion-pair in a molecular capsule.



942

Optimizing the reversibility of hydrazone formation for dynamic combinatorial chemistry

Régis Nguyen and Ivan Huc*

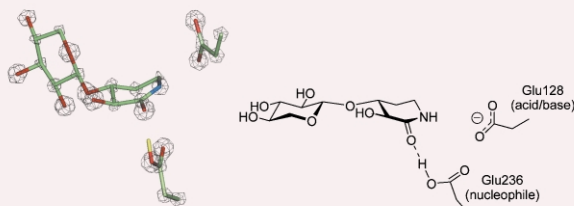


Hydrazone formation from hydrazines bearing electron withdrawing groups, and aromatic or aliphatic aldehydes form and hydrolyse rapidly in water at neutral pH.

944

A xylobiose-derived isofagomine lactam glycosidase inhibitor binds as its amide tautomer

Tracey Gloster, Spencer J. Williams, Chris A. Tarling, Shirley Roberts, Claude Dupont, Pascalé Jodoin, François Shareck, Stephen G. Withers and Gideon J. Davies*

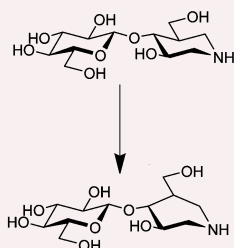


The atomic-resolution structure of the xylobiose-derived isofagomine lactam in complex with the xylanase Xyn10A from *Streptomyces lividans* reveals that the lactam is bound as the amide tautomer, with “reversed” protonation-states for nucleophile and acid–base.

946

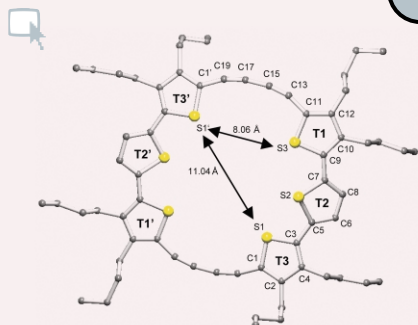
Distortion of a cellobio-derived isofagomine highlights the potential conformational itinerary of inverting β-glucosidases

Annabelle Varrot, James Macdonald, Robert V. Stick, Gavin Pell, Harry J. Gilbert and Gideon J. Davies*



A cellobio-derived isofagomine is distorted to an unusual ^{2.5}B (boat) conformation upon binding to the cellobiohydrolase Cel6A from *Humicola insolens*. This highlights the potential conformational agenda harnessed by this enzyme and illustrates the potential of conformationally-locked glycosidase inhibitors to target specific enzyme classes.

948

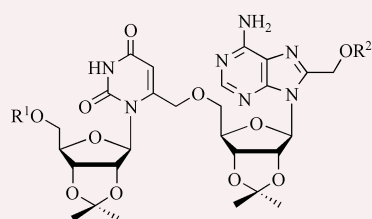


C–C bond formation through oxidatively induced elimination of platinum complexes—A novel approach towards conjugated macrocycles

Gerda Fuhrmann, Tony Debaerdemaeker and Peter Bäuerle*

The effective synthesis of conjugated macrocycles is achieved by self-assembly of conjugated oligomers and transition metal complexes to a metalla-macrocycle and subsequent C–C bond formation.

950



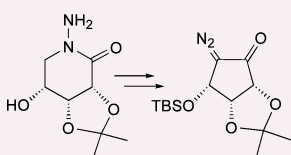
$$K_a = 277\text{--}3222 \text{ M}^{-1}$$

Oligonucleosides with a nucleobase-including backbone; synthesis and self-association of novel dinucleotide analogues

Andrew J. Matthews, Punit K. Bhardwaj and Andrea Vasella*

The first strongly-associating representatives of oligonucleotide analogues with a nucleobase-including backbone have been prepared and characterized.

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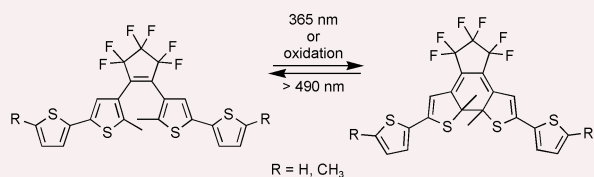


Cyclopentanes from *N*-amino-glyconolactams. A synthesis of mannostatin A

Guixian Hu, Martin Zimmermann, Chepuri Venkata Ramana and Andrea Vasella*

Oxidation of *N*-amino-ribonolactam with lead tetraacetate yields two cyclopentanes; the major product was transformed into the α -mannosidase inhibitor mannostatin A.

954

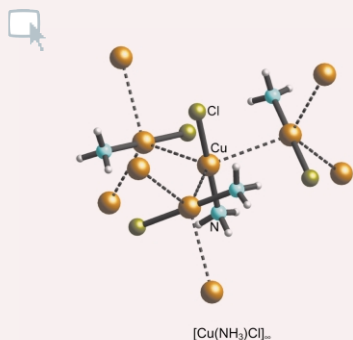


Electrochemically induced ring-closing of photochromic 1,2-dithienylcyclopentenes

Andrea Peters and Neil R. Branda*

An unprecedented combination of photochromism and electrochromism is observed for two 1,2-bis(dithienyl)cyclopentene derivatives; the ring-opening reactions are photochemically driven while the ring-closing reactions can be triggered by electrochemical oxidation.

956



One- and three-dimensional infinite arrays of Cu(I) ions exhibited by [Cu(NH₃)₂]Br and [Cu(NH₃)Cl] in the solid state

Günter Margraf, Jan W. Bats, Michael Bolte, Hans-Wolfram Lerner and Matthias Wagner*

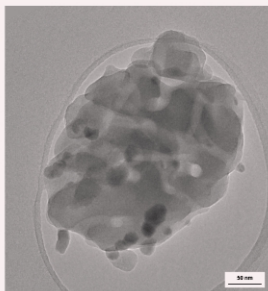
The complexes [Cu(NH₃)Cl] and [Cu(NH₃)₂]Br have been characterized by X-ray crystallography. Both compounds reveal comparatively short Cu...Cu contacts of 2.979(1) Å and 2.931(1) Å, respectively, which are neither ligand enforced nor due to electrostatic attraction.

958

TEM stereo-imaging of mesoporous zeolite single crystals

Astrid Boisen,* Iver Schmidt, Anna Carlsson, Søren Dahl, Michael Brorson and Claus J. H. Jacobsen

Mesoporous zeolite single crystals with intracrystalline mesopores and metal oxide particles located in the zeolite mesopore are characterized by direct TEM stereo-imaging.

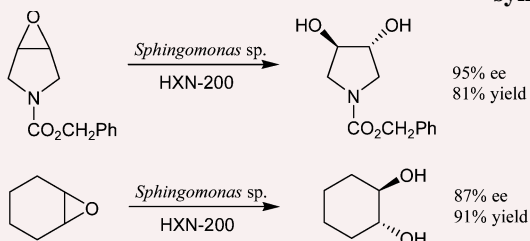


960

Highly enantioselective hydrolysis of alicyclic meso-epoxides with a bacterial epoxide hydrolase from *Sphingomonas* sp. HXN-200: simple syntheses of alicyclic vicinal *trans*-diols

Dongliang Chang, Zunsheng Wang, Maarten F. Heringa, Renato Wirthner, Bernard Witholt and Zhi Li*

High yielding and highly enantioselective hydrolysis of alicyclic meso-epoxides to form the corresponding vicinal *trans*-diols has been achieved, for the first time, with a bacterial epoxide hydrolase.

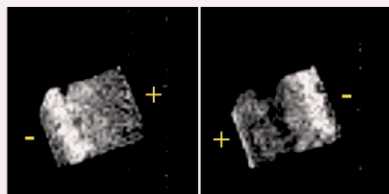


962

***In situ* magnetic resonance imaging of electrically-induced water diffusion in a Nafion ionic polymer film**

Richard T. Baker,* Leila Naji, Karen Lochhead and John A. Chudek

Deployment of a functioning electrochemical cell inside a Magnetic Resonance Imaging instrument to image the electrically-induced diffusion of water through a Li^+ ion-exchanged Nafion film as T_2 and proton density maps.

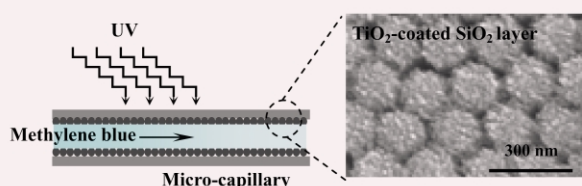


964

Modified micro-space using self-organized nanoparticles for reduction of methylene blue

Xianying Li, Hongzhi Wang, Kouzou Inoue, Masato Uehara, Hiroyuki Nakamura,* Masaya Miyazaki, Eiichi Abe and Hideaki Maeda*

The photoreduction of methylene blue by TiO_2 was significantly improved by using self-organized TiO_2 -coated SiO_2 catalyst layered on the inner surface of a microcapillary channel.

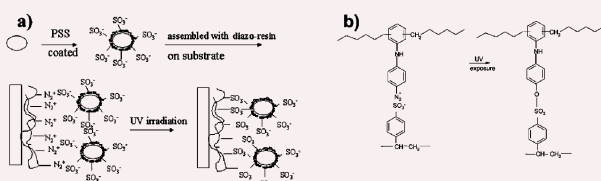


966

Fabrication of a stable inorganic–organic hybrid multilayer film with uniform and dense inorganic nanoparticle deposition

Xurong Xu, Joong Tark Han and Kilwon Cho*

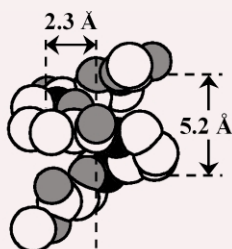
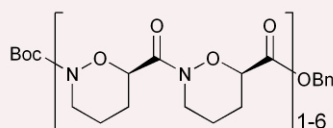
Construction of a stable inorganic–organic hybrid multilayer film with homogeneous and dense inorganic nanoparticle deposition was achieved by coating ZrO_2 nanoparticles with poly(4-sodium styrenesulfonate) (PSS) and irradiating multilayer film assembled from PSS-coated ZrO_2 nanoparticles and a diazo-resin (DR).



968

De novo design of non-hydrogen-bonded helical pseudopeptides composed of oxanipeptic acid oligomers

Myung-ryul Lee, Kwang-Yon Kim, Ung-In Cho, Doo Wan Boo* and Injae Shin*

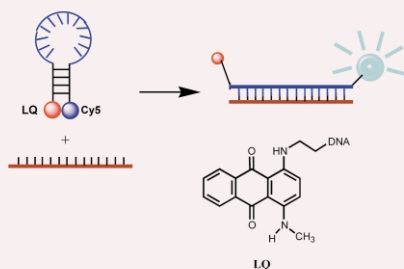


Ab initio calculation and circular dichroism experiments reveal that Oxa-oligomers adopted pronounced non-hydrogen-bonded helical structures.

970

A new dark quencher for use in genetic analysis

Jonathan P. May, Lynda J. Brown, Ivo Rudloff and Tom Brown*

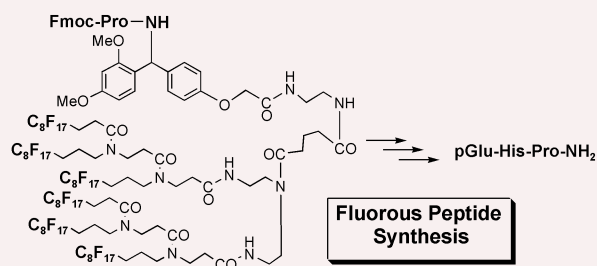


A novel, long-wavelength, non-fluorescent quencher (LQ) has been incorporated at the 3' and 5'-termini of oligonucleotides and used in Molecular beacons, efficiently quenching the long wavelength fluorophore Cy5.

972

A novel peptide synthesis using fluororous chemistry

Mamoru Mizuno, Kohtaro Goto, Tsuyoshi Miura, Daisuke Hosaka and Toshiyuki Inazu*

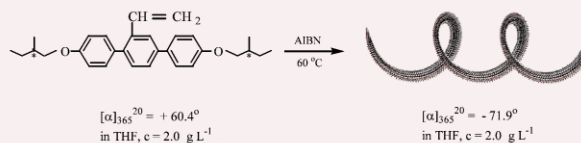


New fluororous supports for peptide synthesis have been prepared. A bioactive peptide TRH was easily synthesized by an Fmoc strategy using the benzhydryl-type fluororous support with fluororous chemistry.

974

A free radical initiated optically active vinyl polymer with memory of chirality after removal of the inducing stereogenic center

Zhenning Yu, Xinhua Wan,* Hailiang Zhang, Xiaofang Chen and Qifeng Zhou

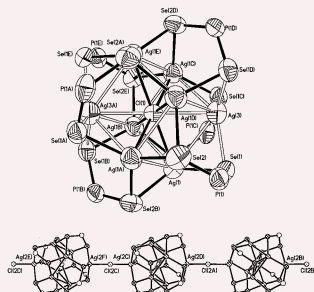


Free radical polymerization of (+)-2,5-bis[4'-((S)-2-methylbutyloxy)phenyl]styrene yields an optically active polymer with memory of chirality after the removal of the initial chiral information in the side group of the monomer.

976

Ag₈Cl₂[Se₂P(OEt)₂]₆: A rare example containing a combination of discrete clusters and chains

C. W. Liu,* Chiu-Mine Hung, Hsien-Chung Haia, Ben-Jie Liaw, Lin-Shu Liou, Yi-Fang Tsai and Ju-Chun Wang

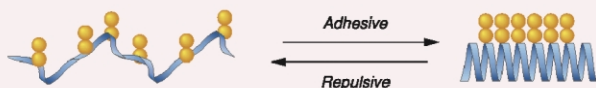


A compound containing a combination of discrete cluster, {Ag₈Cl[Se₂P(OEt)₂]₆}Cl, and chains, {Ag₈Cl[Se₂P(OEt)₂]₆Cl}_n, is characterized for the first time. This phenomenon is due to one of the silver atoms on the threefold rotational axis disordered in two positions.

978

Glyco-helix: parallel lactose-lactose interactions stabilize an α -helical structure of multi-glycosylated peptide

Teruaki Hasegawa and Tomikazu Sasaki*

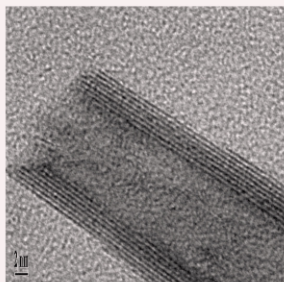


Glyco-helix is designed as a novel model system to investigate *cis* carbohydrate-carbohydrate interactions. Adhesive Lac-Lac interactions stabilize α -helix of Lac-peptide in the presence of fluorinated alcohols, but no such an interaction was observed in GlcNAc-peptide.

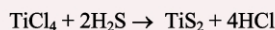
980

Low-temperature synthesis of titanium disulfide nanotubes

Jun Chen,* Suo-Long Li, Zhan-Liang Tao and Feng Gao



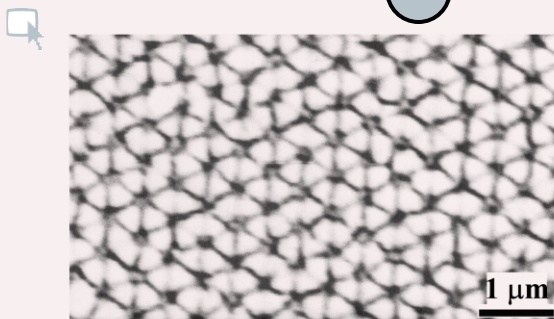
A low-temperature gas reaction at 450 °C was used to synthesize TiS_2 nanotubes, which were characterized by XRD, XPS, TEM-HRTEM, and BET.



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Polymer nanosphere lithography: fabrication of an ordered trigonal polymeric nanostructure

Dong Kee Yi and Dong-Yu Kim*

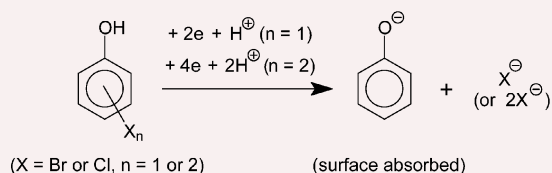


PS-*co*-PDVB colloids were selectively dissolved to fabricate trigonal or rod-like polymeric nanostructures with order and the trigonal arrays became more evident after Ar^+ ion etching.

984

In situ FTIR studies on the electrochemical reduction of halogenated phenols

Raghuram Chetty, Paul A. Christensen and Bernard T. Golding

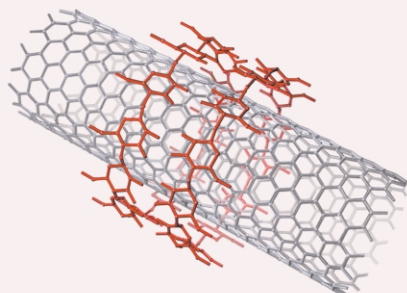


Electrochemical reduction of mono- and di-substituted chloro- and bromo-phenols at palladised titanium in an aqueous electrolyte gave surface-bound phenolate *via* a common intermediate. These observations are relevant to the remediation of toxic halogenated streams.

986

Water solubilization, determination of the number of different types of single-wall carbon nanotubes and their partial separation with respect to diameters by complexation with η -cyclodextrin

Helena Dodziuk,* Andrzej Ejchart, Waldemar Anczewski, Haruhisa Ueda, Elena Krinichnaya, Grygoriy Dolgonos and Wlodzimierz Kutner*

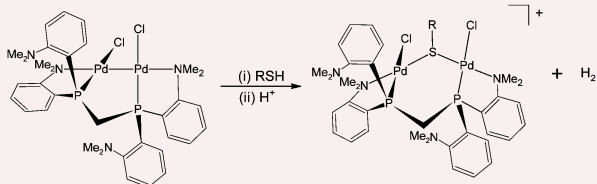


Complexation of single-wall carbon nanotubes with 12-membered cyclodextrins enables not only their solubilization in water but also their partial separation with respect to diameters and determination of the number of nanotubes on the basis of NMR spectra.

988

Diastereoselective formation of a dipalladium(I) complex supported by a bridging tetradentate ligand, and oxidative addition of RS–H across a phosphine-bridged Pd^I–Pd^I bond

S. Jo Ling Foo, Nathan D. Jones, Brian O. Patrick and Brian R. James*

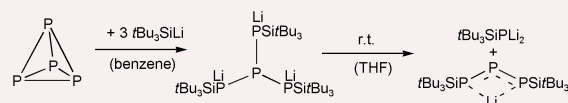


Bonding *via* a μ -*N,P,P,N* moiety of a potentially P_2N_4 -hexadentate gives access to a Pd₂ complex supported by a tetradentate ligand; the complex adds thiols to form a hydrido(thiolato) that with acid gives H₂ and a bridged-thiolate complex.

990

A novel type of phosphide: synthesis and X-ray crystal structure analysis of (tBu₃Si)₃P₄Li₃

Hans-Wolfram Lerner,* Matthias Wagner and Michael Bolte

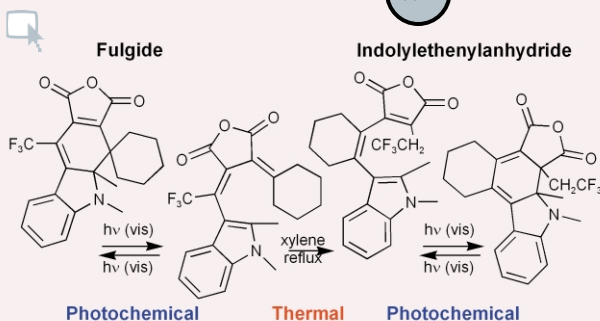


The reaction of 3 equivalents of the silanide *t*Bu₃SiLi with P₄ in benzene leads to the tetraphosphide (tBu₃Si)₃P₄Li₃ which features a dimer in the solid state and can be transformed into the unsaturated triphosphide (tBu₃Si)₂P₃Li and the monophosphide *t*Bu₃SiPLi₂.

992

Thermolysis of fluorinated cycloalkylidene fulgides yields a new class of photochromic compounds

Mason A. Wolak, Nathan B. Gillespie, Robert R. Birge and Watson J. Lees*

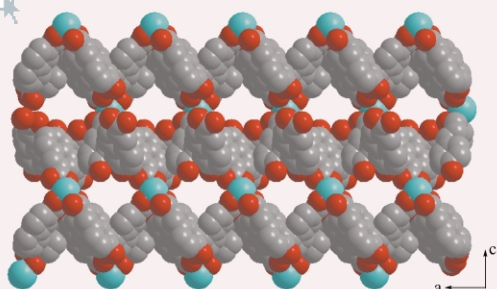


A new class of photochromic compounds, the indolylethenylanhydrides is generated by the thermal rearrangement of another class of photochromic compounds, the fulgides. The indolylethenylanhydrides show enhanced thermal and photochemical stability.

994

Homochiral 3D open frameworks assembled from 1- and 2-D coordination polymers

Yong Cui, Helen L. Ngo, Peter S. White and Wenbin Lin*

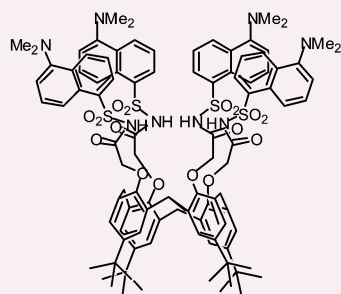


Homochiral 3D open frameworks based on enantiopure atropisomeric dicarboxylic acid bridging ligands have been hierarchically assembled via hydrogen bonding of 1- and 2-D coordination polymeric structures, and retain their framework structures upon the removal of included guest molecules. These homochiral solids provide interesting structural models for the rational design of chiral zeolitic materials.

996

A highly sensitive and selective fluorescent molecular sensor for Pb(II) based on a calix[4]arene bearing four dansyl groups

Rémi Métivier, Isabelle Leray* and Bernard Valeur

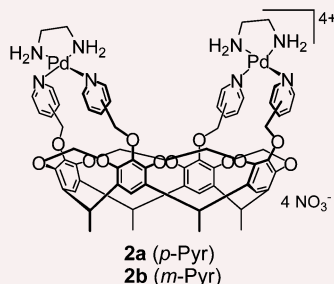


A new fluorescent molecular sensor consisting of a calix[4]arene bearing four dansyl groups shows a high binding efficiency and selectivity for lead with a detection limit of 4 μ g L⁻¹.

998

Water-soluble supramolecular bowls formed by intra-clipping of resorcin[4]arene-based ligands with Pd(II) ions

Seong Jin Park, Dong Mok Shin, Shigeru Sakamoto, Kentaro Yamaguchi, Young Keun Chung, Myoung Soo Lah and Jong-In Hong*

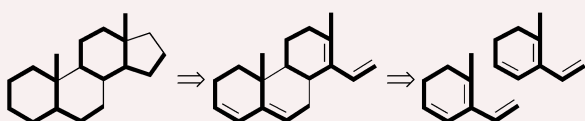
Bowl-shaped superstructures have been constructed by intra-clipping of resorcin[4]arene derivatives with two equivalents of (en)Pd(NO₃)₂ in water.

1000

The role of isomorphism in synthetic analysis. Pruning the search tree by finding disjoint isomorphic substructures

Steven H. Bertz* and Toby J. Sommer

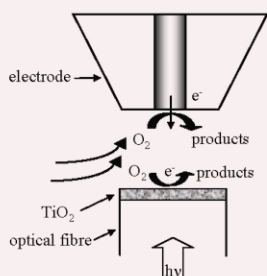
Finding disjoint isomorphic substructures leads to efficient disconnections and reflexive synthetic routes, whether or not there is symmetry in the target molecule, thus pruning the retrosynthetic search tree.



1002

Direct observation of oxygen depletion and product formation during photocatalysis at a TiO₂ surface using scanning electrochemical microscopy

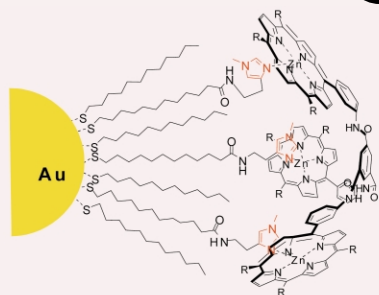
Sofia M. Fonseca, Anna L. Barker, Samina Ahmed, Terence J. Kemp and Patrick R. Unwin*

Significant depletion of O₂ has been detected quantitatively, for the first time, near illuminated TiO₂ surfaces under photomineralisation conditions.

1004

Multivalent recognition of bis- and tris-Zn-porphyrins by *N*-methylimidazole functionalized gold nanoparticles

Gianluca Fantuzzi, Paolo Pengo, Rosa Gomila, Pablo Ballester, Christopher A. Hunter, Lucia Pasquato* and Paolo Scrimin*

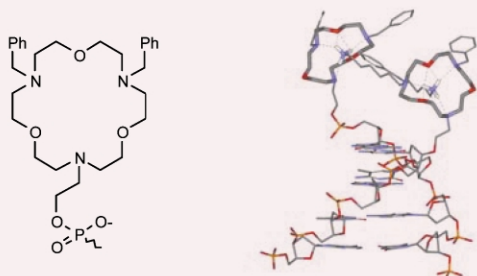
N-Methylimidazole-functionalized gold nanoparticles behave as multivalent ligands for porphyrin arrays with an increase in binding strength of up to three order of magnitude with respect to a monovalent system.

1006

A substituted triaza crown ether as a binding site in DNA conjugates

Stefan Vogel, Katja Rohr, Otto Dahl and Jesper Wengel*

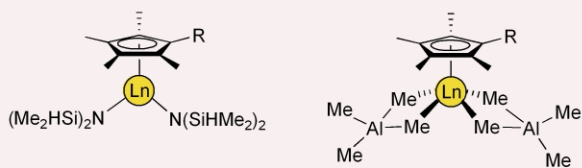
Synthesis of an asymmetrically substituted triaza crown ether, its incorporation into the 3'-end and 5'-end of nine-mer oligonucleotides, and the influence of various alkanediamine ligands on duplex thermostabilities are reported.



1008

High tetraalkylaluminate fluxionality in half-sandwich complexes of the trivalent rare-earth metals

Reiner Anwander,* Michael G. Klimpel, H. Martin Dietrich, Dmitry J. Shorokhov and Wolfgang Scherer

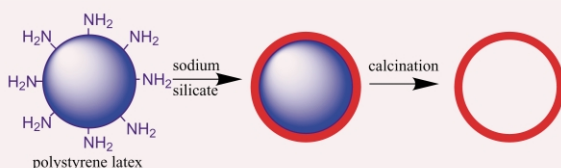


Amine and amide elimination reactions via successive addition of cyclopentadiene and trimethylaluminium to $\text{Ln}[\text{N}(\text{SiHMe}_2)_2](\text{THF})_2$ afford mono(cyclopentadienyl) bis(aluminate) complexes of the smaller rare-earth metal centres in high yield, featuring an all-carbon metal coordination.

1010

Versatile synthesis of nanometer sized hollow silica spheres

Jeroen J. L. M. Cornelissen, Eric. F. Connor, Ho-Cheol Kim, Victor Y. Lee, Teddie Magibitang, Philip M. Rice, Willi Volksen, Linda K. Sundberg and Robert D. Miller*

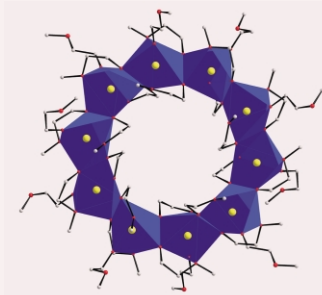


Silica-coated spheres could be prepared by the controlled precipitation of silicic acid on functionalized polystyrene latexes. After removal of the polymer core by calcination, nanometer sized hollow silica spheres were formed.

1012

Self assembly, structure and properties of the decanuclear lanthanide ring complex, $\text{Dy}_{10}(\text{OC}_2\text{H}_4\text{OCH}_3)_{30}$

L. Gunnar Westin,* Mikael Kritikos and Andrea Caneschi

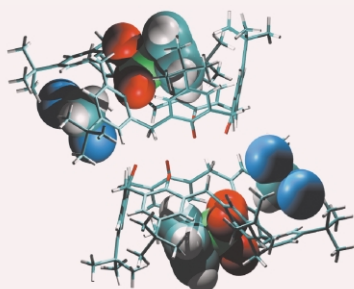


Self assembly produced high yields of the lanthanide ring complex $\text{Dy}_{10}(\text{OC}_2\text{H}_4\text{OCH}_3)_{30}$, the largest lanthanide ring known, characterized by X-ray diffraction methods and by magnetic susceptibility as a function of temperature.

1014

Selective synthesis of conformationally restricted mono-cyclopentadienyl titanium(IV) complexes of *p*-^tBu-calix[6]arene

Antonella J. Petrella,* Nicholas K. Roberts, Donald C. Craig, Colin L. Raston and Robert N. Lamb

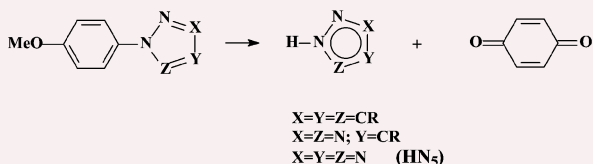


Reaction of *p*-^tBu-calix[6]arene with potassium metal in methanol followed by $[\text{TiCp}_2\text{Cl}_2]$ affords novel mononuclear and binuclear monocyclopentadienyl titanium(IV) complexes, both having the same inverted double cone conformation with a Cp in one of the cavities, in both complexes.

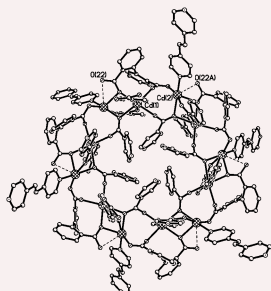
1016

First generation of pentazole (HN_5 , pentazolic acid), the final azole, and a zinc pentazolate salt in solution: A new *N*-dearylation of 1-(*p*-methoxyphenyl) pyrazoles, a 2-(*p*-methoxyphenyl) tetrazole and application of the methodology to 1-(*p*-methoxyphenyl) pentazole

R. N. Butler,* John C. Stephens and Luke A. Burke



A new *N*-dearylation of *N*-*p*-methoxyphenyl azoles, involving phenyl–nitrogen bond cleavage, has been applied to examples of the pyrazole, tetrazole and pentazole rings.



A new type of three-dimensional framework constructed from dodecanuclear cadmium(II) macrocycles

Ruihu Wang, Maochun Hong,* Junhua Luo, Rong Cao and Jiabao Weng

A new type of three-dimensional framework based on dodecanuclear cadmium(II) macrocycles was prepared by the hydrothermal reaction and *in situ* synthesis of pya from dpe precursor.

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