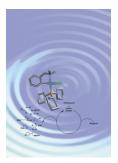


CHEMICAL COMMUNICATIONS • www.rsc.org/chemcomm





#### Cover (far left)

A palladacyclic pre-catalyst, and the range of C–C and C–heteroatom bond-forming reactions mediated by such pre-catalysts. Courtesy of Ann Bingham and Simon Coles of the EPSRC crystallography service, Southampton.

#### Inside cover (left)

A slice cut through a  $BaF_2$  layer, depicting the nanopolycrystalline  $BaF_2$ .

# contents

#### **FOCUS ARTICLE**



Nonenzymatic peptide-based catalytic asymmetric phosphorylation of inositol derivatives

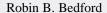
Bianca R. Sculimbrene, Adam J. Morgan and Scott J. Miller\*

Structurally distinct peptide-based catalysts that mediate asymmetric phosphorylation reactions of *meso* derivatives of *myo*-inositol have recently been discovered, and applied to the total synthesis of the enantiomeric I-1P and I-3P targets

#### FEATURE ARTICLE







This article outlines the major role that palladacyclic pre-catalysts have played in a range of C–C and C–heteroatom bond forming reactions, including a discussion on the likely nature of the true active catalysts produced *in situ*.



#### EATURE ARTICLE



Recent developments in the non-cyclopentadienyl organometallic and related chemistry of scandium

Philip Mountford\* and Benjamin D. Ward



Up to the early to mid 1990s the organometallic chemistry of scandium was dominated by cyclopentadienyl derivatives. This present article highlights advances in the synthesis and reactivity of non-cyclopentadienyl organometallic and related compounds of scandium. The graphic gives an illustration of the range of compounds mentioned in this article

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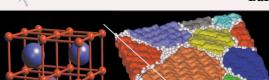
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## Synthesis, structure and ionic conductivity in nanopolycrystalline BaF<sub>2</sub>/CaF<sub>2</sub> heterolayers

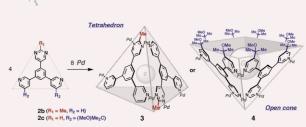


Dean C. Sayle,\* James A. Doig, Stephen C. Parker and Graeme W. Watson

The calculated conductivity of nano-polycrystalline  $BaF_2/CaF_2$  heterolayers is considerably higher compared with the component materials. We suggest that grain-boundary diffusion is the key to fast ion conductivity in these systems.

1808

## Side chain-directed assembly of triangular molecular panels into a tetrahedron $\nu s$ . open cone



Michito Yoshizawa, Muneki Nagao, Kazuhiko Umemoto, Kumar Biradha, Makoto Fujita,\* Shigeru Sakamoto and Kentaro Yamaguchi

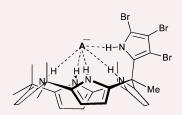
Despite their structural similarity, triangular tetradentate ligands  ${\bf 2b}$  and  ${\bf 2c}$  experience different assembly pathways on complexation with (en)Pd(NO<sub>3</sub>)<sub>2</sub> to give  $M_8L_4$  tetrahedral (3) and open cone (4) structures, respectively.

(1810

#### Pentapyrrolic calix[4]pyrrole

Colin N. Warriner, Philip A. Gale,\* Mark E. Light and Michael B. Hursthouse

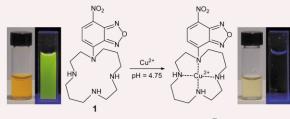
A calix[4]pyrrole with a pendant 2,3,4-trisbromopyrrole attached directly to a *meso*-carbon shows enhanced anion complexation properties relative to *meso*-octamethylcalix[4]pyrrole and displays a considerably enhanced affinity for carboxylates.



1812

#### A two-channel molecular dosimeter for the optical detection of copper(II)

Massimo Boiocchi, Luigi Fabbrizzi,\* Maurizio Licchelli, Donatella Sacchi, Miguel Vázquez and Cristina Zampa



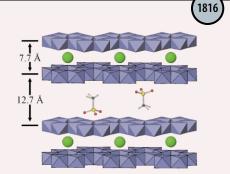
An orange-to-yellow colour change and quenching of the green fluorescence allows selective detection of Cu<sup>2+</sup> in aqueous solution, following metal incorporation by system 1, in which a push–pull chromogenic–fluorogenic fragment is integrated into a tetra-aza macrocycle.

T N-H - N C N N

## Drug-induced stabilisation of a mismatched C-T base pair in a DNA hairpin

Cathal T. Gallagher and Mark S. Searle\*

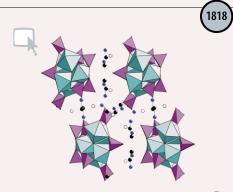
The anthracycline antibiotic nogalamycin is shown by NMR to specifically recognise and stabilise a C-T mismatched base pair within the context of a DNA loop.



The formation of ordered heterostructures during the intercalation of phosphonic acids into a layered double hydroxide

Gareth R. Williams, Alexander J. Norquist and Dermot O'Hare\*

Almost unprecedented ordered second stage heterostructures were observed as intermediate phases in the intercalation of alkylphosphonic acids into a layered double hydroxide.



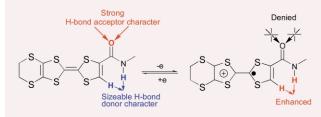
Synthesis and structure determination from powder data of the first organically templated tin(IV) phosphate: MIL-76 or  $Sn_3^{IV}O_2(H_2O)(HPO_4)_4 \cdot \{H_2N-C_2H_4-NH_2\}_{2.5.}\{H_2O\}_2$ 

Christian Serre and Gérard Férey

The first organically templated tin(IV) phosphate has been characterised from Xray powder data. Its one-dimensional structure is built up from trimers of tin(IV) octahedra and phosphate groups interacting with free template and water moieties.



Interdependence of redox state, hydrogen bonding, anion recognition and charge partition in crystals of (EDT-TTF-CONHMe)<sub>6</sub> [Re<sub>6</sub>Se<sub>8</sub>(CN)<sub>6</sub>] (CH<sub>3</sub>CN)<sub>2</sub>(CH<sub>2</sub>Cl<sub>2</sub>)<sub>2</sub>



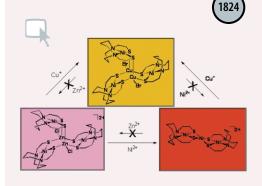
Stéphane A. Baudron, Patrick Batail,\* Carme Rovira, Enric Canadell and Rodolphe Clérac

Upon one-electron oxidation, the carbonyl oxygen atom of EDT-TTF-CONHMe is deactivated with respect to its hydrogen bond acceptor character while the donor character of the N-H and C-H is enhanced.

# Traceless Fischer indole synthesis on the solid phase reduction

Claudia Rosenbaum, Catherine Katzka, Andreas Marzinzik and Herbert Waldmann\*

> The Fischer indole synthesis is employed as the key step for the development of a traceless safety catch indole synthesis on a solid support.



Capture of Ni<sup>II</sup>, Cu<sup>I</sup> and Zn<sup>II</sup> by thiolate sulfurs of an N<sub>2</sub>S<sub>2</sub>Ni complex: A role for a metallothiolate ligand in the acetyl-coenzyme A synthase active site

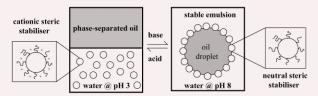
Melissa L. Golden, Marilyn V. Rampersad, Joseph H. Reibenspies and Marcetta Y. Darensbourg\*

The metal binding affinity of an (N<sub>2</sub>S<sub>2</sub>)Ni bridging metallothiolate ligand are established:  $(Zn^{2+} < Ni^{2+} < Cu^+)$ . Implications for the controversy regarding the metal composition, [NiM], of acetyl-coenzyme A active site are presented.



Use of sterically-stabilised polystyrene latex particles as a pH-responsive particulate emulsifier to prepare surfactant-free oil-in-water emulsions

J. I. Amalvy, S. P. Armes,\* B. P. Binks,\* J. A. Rodrigues and G-F. Unali



A pH-responsive, sterically-stabilised polystyrene latex is used as a particulate emulsifier for oil-in-water emulsions; demulsification occurs on lowering the solution pH and the emulsion is reformed on pH cycling.



1830

A unique series of dinuclear transition metal—polyradical complexes with a *m*-phenylenediamine spacer and their catalytic reactivity

Soumen Mukherjee, Eva Rentschler, Thomas Weyhermüller, Karl Wieghardt and Phalguni Chaudhuri\*

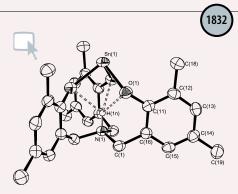
A series of dinuclear transition metal complexes with either six or four iminosemiquinone radicals, in which the metal centres are separated by a distance of ~6.8 Å, together with their catalytic reactivity is reported.



Effects of trace metals and organic additives on porosity and dielectric constant of high purity mesoporous silica films

Jerome C. Birnbaum, Glen E. Fryxell, Xiaohong Li, Chris A. Coyle, Glen C. Dunham and Suresh Baskaran\*

The beneficial effects that alkali metal and tetra-alkyl ammonium salt additions to molecularly templated silica sols have on the resulting mesoporous silica film are described. Films, formed from evaporative-coating methods, using the doped sols exhibited significant improvement in porosity, film surface uniformity, and dielectric constant.



Isolation and characterisation of transition and main group metal complexes supported by hydrogen-bonded zwitterionic polyphenolic ligands

Matthew G. Davidson, Cheryl L. Doherty, Andrew L. Johnson and Mary F. Mahon

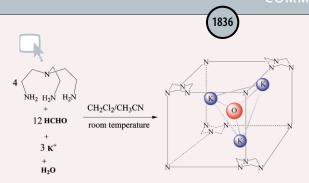
Reaction of  $Zr(O^iPr)_4$  or  $Sn[N(SiMe_3)_2]_2$  with the tris-phenol amine ligand  $H_3L^{(Me/Me)}$  results in the formation of zirconium or tin complexes containing the new  $C_3$ -symmetric zwitterionic ammonium-trisphenolate ligand  $HL^{2-}$ .

DAC R1 CO<sub>2</sub>Et NHBoc (E)-alkene dipeptide isosteres R2 HO NHBoc NHBoc CO<sub>2</sub>Et γ-amino acids

Application of samarium diiodide (SmI<sub>2</sub>)-induced reduction of  $\gamma$ -acetoxy- $\alpha,\beta$ -enoates with  $\alpha$ -specific kinetic electrophilic trapping for the synthesis of amino acid derivatives

Akira Otaka,\* Akira Yukimasa, Junko Watanabe, Yoshikazu Sasaki, Sinya Oishi, Hirokazu Tamamura and Nobutaka Fujii

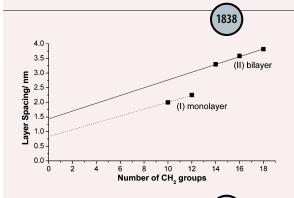
Samarium diiodide (SmI<sub>2</sub>)-induced reductive formation of dienolates from  $\gamma$ -acetoxy- $\alpha$ , $\beta$ -enoates followed by kinetic electrophilic trapping was successfully applied to the synthesis of various amino acid derivatives.



## A novel pyramidal multiple alkali metal cluster $[K_3(H_2O)]^{3+}$ stabilized within a capsule containing 16-nitrogen donors

Zhang Bing-guang, Guo Dong, Duan Chun-ying,\* Cai Ping and Meng Qing-jin\*

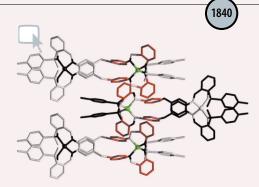
A novel pyramidal multiple alkali metal cluster  $[K_3(H_2O)]^{3+}$  stabilized within a capsule containing 16-nitrogen donors.



## A series of long-chain lamellar hydrated copper( $\Pi$ ) alkylsulfonates with different chain molecular assemblies

Seong-Hun Park and Cheol Eui Lee\*

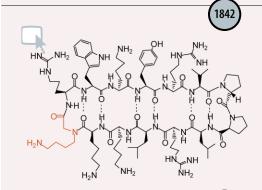
Group (I) materials with smaller carbon numbers are composed of interdigitated alkyl chains, whereas group (II) materials are of a non-interdigitated bilayer structure.



#### A 3D network of helicates fully assembled by $\pi$ -stacking interactions

Miguel Vázquez, Angelo Taglietti, Dante Gatteschi,\* Lorenzo Sorace, Claudio Sangregorio, Ana M. González, Marcelino Maneiro, Rosa M. Pedrido and Manuel R. Bermejo\*

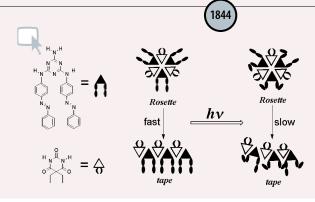
The reported neutral dihelicate forms a unique 3D network in the solid state due to  $\pi$ -stacking interactions, which are responsible for intermolecular antiferromagnetic coupling between Cu(II) ions: this phenomenon is reported for the first time for this class of supramolecular arrays.



## A family of macrocyclic antibiotics with a mixed peptide-peptoid ß-hairpin backbone conformation

Sasalu C. Shankaramma, Kerstin Moehle, Sonya James, Jan W. Vrijbloed, Daniel Obrecht and John A. Robinson\*

Novel cationic, macrocyclic peptidomimetics are described that adopt a stable  $\beta$ -hairpin conformation in aqueous solution, and show antimicrobial activity against Gram +ve and Gram —ve bacteria.



## Photoresponsive melamine barbiturate hydrogen-bonded assembly

Shiki Yagai,\* Takashi Karatsu and Akihide Kitamura\*

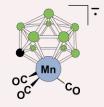
Hydrogen-bonded assembly between a melamine–azobenzene conjugate and barbiturate is reported. In CHCl<sub>3</sub>, transformation of the soluble *rosette* assemblies into the insoluble *tapelike* assemblies was suppressed drastically *via* photoisomerization of azobenzene units.



The seventeen- and eighteen-electron metallacarbaboranes  $[1,1,1-(CO)_3-2-Ph-closo-1,2-MnCB_9H_9]^{n-}$  (n=1,2): a structurally characterized, redox-related pair

Shaowu Du, Robert D. Farley, Jeremy N. Harvey, John C. Jeffery, Jason A. Kautz, John P. Maher, Thomas D. McGrath, Damien M. Murphy, T. Riis-Johannessen and F. Gordon A. Stone\*

The unpaired electron in a manganese-carbaborane radical anion is delocalized over the entire cluster.

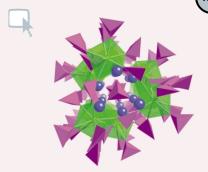


(1848)

## Synthesis and characterization of a channel framework in $K_3Am_3(IO_3)_{12}$ ·HIO $_3$

Wolfgang Runde,\* Amanda C. Bean and Brian L. Scott

The hydrothermal reaction of Am(III) with  $KIO_4$  produced pink crystals of  $K_3Am_3(IO_3)_{12}$ ·HIO<sub>3</sub>. This compound represents the first structurally characterized actinide(III) iodate containing a three-dimensional architecture that has not been observed among lanthanide iodates.

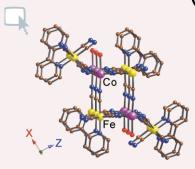


(1850)

## Cyanide-bridged Fe(III)-Co(II) bis double zigzag chains with a slow relaxation of the magnetisation

Luminita Marilena Toma, Rodrigue Lescouëzec, Francesc Lloret, Miguel Julve,\* Jacqueline Vaissermann and Michel Verdaguer

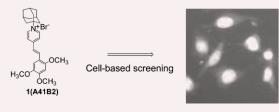
Bis double zigzag chains [[Fe<sup>III</sup>(bipy)(CN)<sub>4</sub>]<sub>2</sub>M<sup>II</sup>(H<sub>2</sub>O)]·MeCN· $\frac{1}{2}$ H<sub>2</sub>O; **1** exhibits intrachain ferromagnetic and interchain antiferromagnetic couplings, slow magnetic relaxation and hysteresis effects which are not associated with a three-dimensional order whereas **2** shows an overall antiferromagnetic coupling.



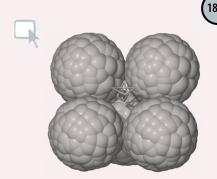
(1852)

## Development of novel cell-permeable DNA sensitive dyes using combinatorial synthesis and cell-based screening

Jae Wook Lee, Michelle Jung, Gustavo R. Rosania and Young-Tae Chang\*



A novel cell-permeable DNA fluorescence sensor was developed based on combinatorially-created styryl dyes and cell-based localization screening.



#### Characterisation of a new 1:1 (C<sub>60</sub>)(CHBr<sub>3</sub>) intercalation complex

Michaele J. Hardie,\* Ralph Torrens and Colin L. Raston\*

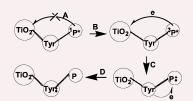
The new 1:1 crystalline intercalation complex  $(C_{60})(CHBr_3)$  has been synthesised and structurally characterised. It has an irregular structure comprised of  $C_{60}$  molecules interspersed with zigzag chains of disordered bromoform molecules.



## Porphyrin capped TiO<sub>2</sub> nanoclusters, tyrosine methyl ester enhanced electron transfer

Junhua Yu, Jingrong Chen, Xuesong Wang,\* Baowen Zhang\* and Yi Cao

The photoinduced electron transfer between neutral titanium dioxide nanoclusters and porphyrin is enhanced by the bridging of tyrosine methyl ester to the two components.

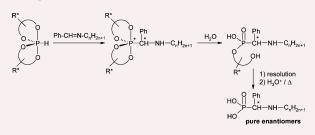


## 1858

## Asymmetric synthesis of ( $\alpha\text{-amino})\text{phosphonic}$ acid amphiphiles using chiral P–H spirophosphoranes

Christophe Déjugnat, Guita Etemad-Moghadam and Isabelle Rico-Lattes\*

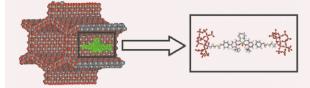
Stereoselective addition reaction between chiral P–H spirophosphoranes and long-chain prochiral aldimines affording, after selective hydrolysis, enantiopure ( $\alpha$ -amino)phosphonic acid amphiphiles in a monoester or free acid form.



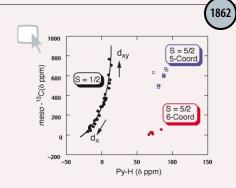
#### (1860)

## Synthesis and catalytic activity of a chiral periodic mesoporous organosilica (ChiMO)

Carlos Baleizão, Bárbara Gigante,\* Debasish Das, Mercedes Alvaro, Hermenegildo Garcia\* and Avelino Corma\*



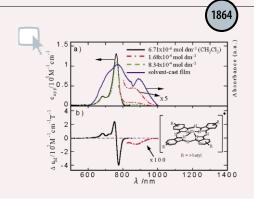
Chiral vanadyl salen complex incorporated in the framework of a MCM-41 is active for the enantioselective catalytic addition of cyanotrimethylsilane to aldehydes.



# Chemical shift of meso-carbon: a powerful probe to determine the coordination structure and electron configuration of ferric porphyrin complexes

Mikio Nakamura,\* Akito Hoshino, Akira Ikezaki and Takahisa Ikeue

*meso*-<sup>13</sup>C chemical shifts serve as a powerful probe to determine the coordination structure and electron configuration of ferric porphyrin complexes



## Spectral properties of a novel antimony(III)-phthalocyanine complex that behaves like J-aggregates in non-aqueous media

Hiroaki Isago\*

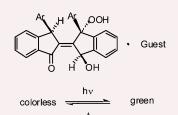
An unusual red-shift of phthalocyanine Q-band upon aggregation in non-aqueous media has been observed for antimony(III) derivative and has been studied by using optical absorption and magnetic circular dichroism spectroscopy.



## Photochromism of the inclusion crystals of a new hydroperoxybiindenylidene

Koichi Tanaka,\* Yohei Yamamoto and Shigeru Ohba

Inclusion crystals of 3,1′-bis(4-fluorophenyl)-1′-hydroperoxy-3′-hydroxy-1′,3′-dihydro-3*H*-[2,2′]biindenyliden-1-one showed photochromic and photomagnetic properties in the solid state.



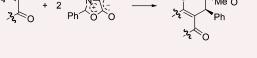


1870

## Regio and diastereoselective lactonisation of enolisable 1,3-dicarbonyls by reaction with mesoionic 1,3-oxazolium-5-olates

Giovanni Grassi,\* Francesco Risitano, Francesco Foti, Massimiliano Cordaro, Giuseppe Bruno and Francesco Nicolò

Reaction of enolisable 1,3-dicarbonyls and N-methyl-1,3-oxazolium-5-olate derivatives provided enol lactones directly in good yield and with excellent regio- and diastereocontrol.



## Amidinyl radicals: new and useful intermediates for the synthesis of imidazolines and imidazoles

Dominique Gennet, Samir Z. Zard\* and Haiwen Zhang

Amidinyl radicals are readily generated from amidoxime benzoates by treatment with a stannane–diazo initiator or with Ni–AcOH and captured by an internal olefin to give the corresponding imidazoline.

## 1872

#### Synthesis and biological evaluation of cytostatin analogues

Laurent Bialy\* and Herbert Waldmann

deprotected

essential

not essential, but beneficial for activity

must be fully

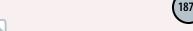
The basic structure–activity relationship (SAR) of PP2A inhibition of the phosphatase inhibitor cytostatin has been derived from the evaluation of a small set of analogues of the natural product.



## Synthesis of the aminocyclopentitol moieties of the hopanoids of *Zymomonas mobilis* and '*Anacystis montana*'

Jose Luis Chiara,\* Isabel Storch de Gracia and Ágatha Bastida

The first synthesis of the cyclopentitol units in bacterial hopanoids  ${\bf 1a}$  and  ${\bf 1b}$  has been accomplished from D-glucosamine. The free cyclitol in  ${\bf 1a}$  shows weak inhibitory activity against  $\alpha$ - and  $\beta$ -glucosidases and  $\alpha$ - and  $\beta$ -galactosidases.



## Barium hexaaluminate nanowhiskers synthesized by novel sol-gel process in reverse micellar media

Prashant K. Sahu, B. D. Kulkarni,\* R. B. Khomane, S. A. Pardhy, U. D. Phalgune, P. Rajmohanan and Renu Pasricha

Barium hexaaluminate (BHA) synthesized by coupling of sol-gel process in reverse microemulsions shows a unique nanowhisker morphology and high surface area, which are retained after calcination at 1300 °C.

1878

# Excitation Emission Emis

#### Pyrene as a fluorescent probe for DNA base radicals

Robert Huber, Torsten Fiebig\* and Hans-Achim Wagenknecht\*

The pH-dependent emission of four pyrene-modified nucleosides provide important information about the acidity or basicity of the DNA base radicals which is relevant to the understanding of charge transport in DNA.

(1880)

## Pore mouth *versus* intracrystalline adsorption of isoalkanes on ZSM-22 and ZSM-23 zeolites under vapour and liquid phase conditions



J. F. Denayer,\* A. R. Ocakoglu, W. Huybrechts, J. A. Martens, J. W. Thybaut, G. B. Marin and G. V. Baron

Adsorption experiments reveal that iso-alkanes adsorb only with their linear tail in the pore entrances of ZSM-23 in vapor and liquid phase conditions.

(1

## Strong optical limiting capability of a triosmium cluster bonded indium porphyrin complex [(TPP)InOs $_3(\mu$ -H) $_2(CO)_9(\mu$ - $\eta^2$ -C $_5$ H $_4$ N)]

Xinhua Zhong, Yaoyu Feng,\* Say-Leong Ong, Jiangyong Hu, Wun-Jern Ng and Zheming Wang

A novel metal-metal bonded indium porphyrin-osmium cluster complex with strong optical limiting capability was first synthesized using the indium porphyrin hydride as precursor and characterized.

OC)<sub>3</sub>OS H OS(CO)<sub>3</sub>

## 1884

## A controllable synthetic route to Cu, Cu<sub>2</sub>O, and CuO nanotubes and nanorods

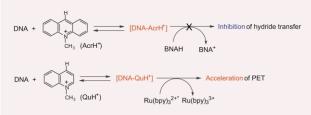
Minhua Cao, Changwen Hu,\* Yonghui Wang, Yihang Guo, Caixin Guo and Enbo Wang

 $Cu(OH)_{4}^{2} \xrightarrow{CTAB} Cu(OH)_{4}^{2} \xrightarrow{-CTA}^{+} \begin{cases} & \text{hydrazine hydrate} \\ & \text{at room temperature} \end{cases} \xrightarrow{Cu \text{ nanotubes/nanorods}} \\ & \text{cu}(OH)_{4}^{2} \xrightarrow{-CTA}^{+} \begin{cases} & \text{glucose} \\ & \text{at room temperature} \end{cases} \xrightarrow{Cu_{2}O \text{ nanotubes/nanorods}} \\ & \text{hydrothermal } 120 \text{ °C} \end{cases}$ 

Reducing  $\text{Cu(OH)}_4^{2-}$  with hydrazine hydrate and glucose in the presence of a structure-directing surfactant at room temperature gave Cu and  $\text{Cu}_2\text{O}$  nanotubes/nanorods, respectively, whereas facile hydrothermal treatment of the  $\text{Cu(OH)}_4^{2-}$  precursor resulted in CuO nanotubes/nanorods.

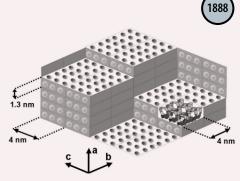


## Reversed effects of DNA on hydride transfer and electron transfer reactions of acridinium and quinolinium ions



Mari Nishimine, Kei Ohkubo, Takashi Komori and Shunichi Fukuzumi\*

DNA inhibits hydride transfer from 1-benzyl-1,4-dihydronicotinamide to the 10-methylacridinium ion (AcrH $^+$ ), whereas DNA accelerates photoinduced electron transfer (PET) from the excited state of Ru(bpy) $_3^{2+}$  (bpy = 2,2'-bipyridine) to the 1-methylquinolinium ion (QuH $^+$ ).



## n-Alkane hydroconversion on Zeogrid and colloidal ZSM-5 assembled from aluminosilicate nanoslabs of MFI framework type

Alexander Aerts, Ward Huybrechts, Sebastien P. B. Kremer, Christine E. A. Kirschhock, Elisabeth Theunissen, Annabel Van Isacker, Joeri F. M. Denayer, Gino V. Baron, Joris W. Thybaut, Guy B. Marin, Pierre A. Jacobs and Johan A. Martens\*

In decane hydroisomerisation, colloidal ZSM-5 and Zeogrid prepared by self-assembly of nanoslabs exhibit molecular shape selectivity different from ZSM-5 obtained by hydrothermal synthesis.



1892

## Synthesis of (pyridin-2-yl)hydrazine conjugates as bifunctional chelates using the Suzuki-Miyaura reaction



Jeffrey B. Arterburn,\* Bj K. Bryant and DaJun Chen

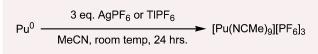
Palladium catalyzed C–C couplings were used to connect (pyridin-2-yl)hydrazine to organic substrates, including a phenylalanine derivative, providing a new method for introducing this important ligand.



ΝH

## Preparation and structures of homoleptic $Pu(\mathrm{III})$ and $U(\mathrm{III})$ acetonitrile salts

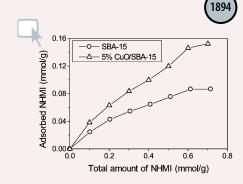
Alejandro E. Enriquez, John H. Matonic, Brian L. Scott and Mary P. Neu\*



N<sup>t</sup>BocN(H)<sup>t</sup>Boc

Nine-coordinate homoleptic acetonitrile solvate complexes of Pu(III) and U(III) ions have been prepared through oxidation of Pu metal suspended in acetonitrile with metal-hexafluorophosphate salts and dissolution of  $UI_3(THF)_4$  in acetonitrile, respectively.





#### Trapping volatile nitrosamines with copper incorporated zeolites

Yang Xu, Zhi-yu Yun, Jian Hua Zhu,\* Jia-hui Xu, Hua-dao Liu, Yi-lun Wei and Kang-jin Hui

Modification of zeolite or mesoporous materials with copper oxide significantly promotes the selectively adsorption of volatile nitrosamines, beneficial for the removal of nitrosamines in cigarette smoke.



Perchloric acid adsorbed on silica gel as a new, highly efficient, and versatile catalyst for acetylation of phenols, thiols, alcohols, and amines

Asit K. Chakraborti\* and Rajesh Gulhane

 $Ar(R)XH \xrightarrow{Ac_2O/HOAc} Ar(R)XCOCH_3$   $X = O, S, NH \qquad (1 \text{ mol } \%) \qquad (75-100\%)$ 

HClO<sub>4</sub>–SiO<sub>2</sub> efficiently catalyzes the acetylation of phenols, thiols, amines, and alcohols at room temperature under solvent free conditions. Excellent results are obtained during the direct acetylation of alcohols with HOAc.



Immobilized heterobimetallic Ru/Co nanoparticle-catalyzed Pauson-Khand-type reactions in the presence of pyridylmethyl formate

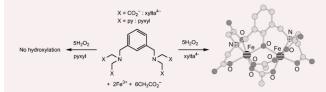
Kang Hyun Park, Seung Uk Son and Young Keun Chung\*



Heterobimetallic Ru/Co nanoparticles immobilized on charcoal were synthesized and used as catalysts in the Pauson–Khand-type reaction in the presence of pyridylmethyl formate instead of carbon monoxide; the catalysts were effective for intra- and intermolecular reactions and easily reused without loss of catalytic activity.



Regioselective hydroxylation of the xylyl linker in a diiron(III) complex having a carboxylate-rich ligand with  $H_2O_2$ 



Hideki Furutachi, Mizue Murayama, Amane Shiohara, Satoshi Yamazaki, Shuhei Fujinami, Akira Uehara, Masatatsu Suzuki,\* Seiji Ogo, Yoshihito Watanabe and Yonezo Maeda

Reaction of a diiron(III) complex having a  $xylta^{4-}$  ligand with  $H_2O_2$  resulted in regioselective hydroxylation of the m-xylyl linker, mimicking the self-hydroxylation of a phenylalanine sidechain found for R2-W-48F/D84E.



Palladium catalysed [2+2+1] intramolecular cycloaddition for the preparation of bicyclo[3.3.0]octa-1.5-dien-3-ones from 1,6-diynes

Ronald Grigg,\* Lixin Zhang, Simon Collard and Ann Keep

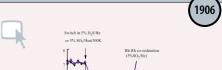
Palladium catalysed [2+2+1] cycloaddition of 1,6-heptadiynes with CO (1 atm) furnishes bicyclo[3.3.0]octa-1,5-dien-3-ones in 30–74% yield. The use of low pressure carbon monoxide and the high density of functionality in the products make them versatile building blocks for further elaboration.



#### $\pi\text{-Stacking}$ interactions in some crystalline cisoid E,E-1,4-diaryl-1,3-but but adienes

Jin Liu,\* Elisia M. Murray and Victor G. Young, Jr

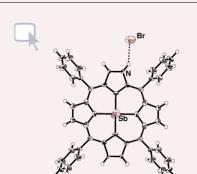
A cisoid E,E-1,4-diperfluorophenyl-1,3-butadiene has been prepared in which offset stacking between perfluorophenyl-perfluorophenyl rings occurs, and faceto-face stacking between phenyl-perfluorophenyl rings is found in crystals of its 1:1 complex with a cisoid E,E-1,4-diphenyl-1,3-butadiene.



Contrasting dynamic responses of supported Rh nanoparticles to  $H_2S$  and  $SO_2$  and subsequent poisoning of NO reduction by  $H_2$ 

Mark A. Newton,\* Andrew J. Dent, Sofia Diaz-Moreno, Steven G. Fiddy, Bhrat Jyoti and John Evans

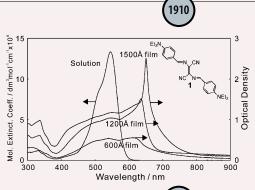
Dispersive EXAFS delineates the dynamic structural response of supported Rh nanoparticles toward  $H_2S$  and  $SO_2$  that underpin the different effects these gases have on selective NO reduction by  $H_2$ .



 $\label{eq:modulation} \begin{tabular}{ll} Modulation of axial coordination in $N$-confused porphyrin-antimony (v) \\ dibromide complex by proton stimulus \\ \end{tabular}$ 

Jia-Cheng Liu, Tomoya Ishizuka, Atsuhiro Osuka and Hiroyuki Furuta\*

The change of the axial bond length in the antimony(v) N-confused tetraphenylporphyrin dibromide complex was demonstrated by protonation and confirmed from X-ray single crystal analyses.



J-Aggregates in vapor deposited films of a bisazomethine dye

Shinya Matsumoto,\* Takashi Kobayashi, Tetsuya Aoyama and Tatsuo Wada

This is the first report of J-aggregate formation of a non-ionic bisazomethine dye in vapor deposited films; this dye allows us to prepare easily large homogeneous and very stable J-aggregate thin films and to investigate intrinsic properties of low-dimensional Frenkel excitons.

1912

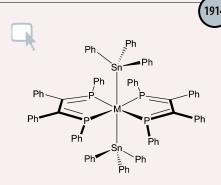
1908

Highly enantioselective catalytic asymmetric hydrogenation of  $\beta\text{-keto}$  esters in room temperature ionic liquids

Helen L. Ngo, Aiguo Hu and Wenbin Lin\*

$$R_1$$
  $R_2$   $R_2$   $R_3$   $R_4$   $R_4$   $R_4$   $R_4$   $R_5$   $R_6$   $R_6$   $R_8$   $R_8$   $R_9$   $R_9$ 

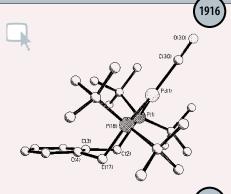
Polar phosphonic acid-derived Ru-BINAP systems were used to catalyze asymmetric hydrogenation of  $\beta$ -keto esters in room temperature ionic liquids (RTILs) with complete conversions and ee values up to 99.3%. The enantioselectivity obtained in RTILs–MeOH is higher than that achieved in MeOH. Both the catalysts and RTILs were recycled *via* simple extraction.



Neutral and dianionic iron and ruthenium 1,4-diphosphabutadiene complexes

Audrey Moores, Nicolas Mézailles, Louis Ricard, François Mathey and Pascal Le Floch\*

Synthesis of dianionic iron and ruthenium complexes is reported. These highly reactive species were trapped with Ph<sub>3</sub>SnCl to yield the corresponding bis(1,4-diphosphabutadiene) complexes. X-Ray structures are presented.



## Synthesis, X-ray characterisation and reactions of a trigonal planar palladium(0) carbonyl complex, (tbpx)PdCO

Ronan M. Bellabarba,\* Robert P. Tooze and Alexandra M. Z. Slawin

The novel complex (tbpx)PdCO, the first example of a structurally characterised sixteen electron, trigonal planar palladium(0) carbonyl complex, was prepared, characterised by NMR spectroscopy and X-ray crystallography, and some unusual aspects of its reactivity were studied.

## 1918

## A facile aminocyclization for the synthesis of pyrrolidine and piperidine derivatives

Zhihui Shao, Jingbo Chen, Yongqiang Tu, Liang Li and Hongbin Zhang\*

furnishes a highly stereoselective protocol for the intramolecular formation of pyrrolidine and piperidine ring containing subunits that are presented in numerous biologically active natural products.

Bromination of an isolated double bond followed by aminocyclization

n = 1, 2; m = 1, 2; R = Protecting group



## Novel nanoporous hybrid organic-inorganic silica containing iminodiethanol chelating groups inside the channel pores

Chunqing Liu, Nathaniel Naismith, Lei Fu and James Economy\*



A novel nanoporous hybrid organic-inorganic silica with covalently bound iminodiethanol chelating groups inside the channel pores has been synthesized and is shown to be very efficient in recovery of germanium and antimony oxides from water.



## Solid-solid transformation mechanism for nanocrystalline sodalite from pillared clay

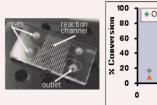
Jin-Ho Choy,\* Sung-Reol Lee, Yang-Su Han, Man Park and Gyeong-Su Park

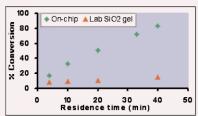


Nanocrystalline sodalite has been synthesized through a solid–solid transformation from a solid gel mixture of  $Al_2O_3$  pillared montmorillonite and hydrated NaOH under mild non-hydrothermal conditions.



## Surface effects in the esterification of 9-pyrenebutyric acid within a glass micro reactor





Monica Brivio, R. Edwin Oosterbroek, Willem Verboom,\* Martijn H. Goedbloed, Albert van den Berg and David N. Reinhoudt\*

Surface phenomena are an important contribution to the "chip effect", leading to higher yields and shorter reaction times, as demonstrated for the acid-catalysed esterification of 9-pyrenebutyric acid within a glass fabricated micro reactor.



Observation of high enantioselectivity for the gas phase hydrogenation of methyl pyruvate using supported Pt catalysts pre-modified with cinchonidine

Matthias von Arx, Nicholas Dummer, David J. Willock, Stuart H. Taylor, Richard P. K. Wells, Peter B. Wells and Graham J. Hutchings\*

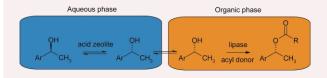
OMe 
$$\frac{H_z}{PU_TAI_zO_3$$
 + cinchonidine  $OMe$   $OME$ 

Supported Pt catalysts, pre-modified with cinchonidine, give high ee using gas phase reactants giving the first clear observation of high enantioselection at the gas/solid interface.

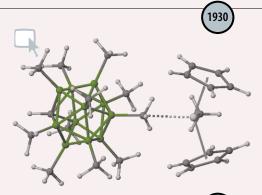


## A zeolite-enzyme combination for biphasic dynamic kinetic resolution of benzylic alcohols

Stijn Wuyts, Karolien De Temmerman, Dirk De Vos and Pierre Jacobs\*



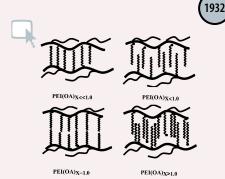
Acid zeolites were found to be very efficient heterogeneous catalysts for racemization of benzylic alcohols in aqueous medium and could be combined in a two-phase system with an enzymatic kinetic resolution resulting in a successful dynamic kinetic resolution (DKR) with a high yield and enantiomeric purity of the resulting product.



## [ $Cp_2ZrMe(12-\mu-Me-1-closo-CB_{11}HMe_{10})$ ]: a transition metal complex of a highly-methylated carborane anion

Michael J. Ingleson, Adam Clarke, Mary F. Mahon, Jonathan P. Rourke and Andrew S. Weller\*

[Cp<sub>2</sub>ZrMe(12- $\mu$ -Me-1-closo-CB<sub>11</sub>HMe<sub>10</sub>)], the first transition metal complex of an highly alkylated carborane anion, is isolated by methide abstraction from Cp<sub>2</sub>ZrMe<sub>2</sub> using [CPh<sub>3</sub>][1-closo-HCB<sub>11</sub>Me<sub>11</sub>] and displays a significant intermolecular Zr···H<sub>3</sub>C interaction in both the solid state and solution.



## Lamellar architecture and crystalline transformation in supramolecular complexes of highly-branched polyethyleneimine-octadecanoic acid

Surong Zhou, Ying Zhao, Yuanli Cai, Dujin Wang\* and Duanfu Xu

Supramolecular complexes of highly-branched polyethyleneimine-octadecanoic acid  $PEI(OA)_x$  in the solid state showed typical lamellar structure, and reversible crystalline phase transformation of side alkyl chains was observed with temperature variable FT-IR spectroscopy.



## A very efficient, copper-free palladium catalyst for the Sonogashira reaction with aryl halides

Denise Méry, Karine Heuzé and Didier Astruc

$$R \xrightarrow{\qquad \qquad } X + H \xrightarrow{\qquad \qquad } R' \xrightarrow{\qquad \qquad } R \xrightarrow{\qquad \qquad } R'$$

The new electron-rich bis-*tert*-butylphosphine palladium(II) complex represented on the scheme catalyses the Sonogashira reaction between aryl halides with acetylenes at room temperature without co-catalyst.

## Rh-Catalyzed one-pot and practical transformation of aldoximes to amides

Soyoung Park, Yoon-aa Choi, Hoon Han, Soon Ha Yang and Sukbok Chang\*

Wilkinson's complex has been found to catalyze the one-pot transformation of aldoximes to the corresponding amides with high selectivity and efficiency under essentially neutral conditions.



in solution

The ion gating effect: using a change in flexibility to allow label free electrochemical detection of DNA hybridisation

J. Justin Gooding,\* Alison Chou, Freya J. Mearns, Elicia (Leh-See) Wong and Kellie L. Jericho

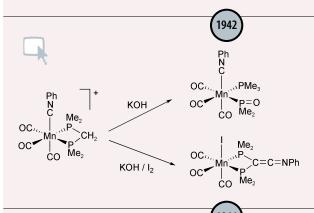
Label-free detection of DNA hybridisation is demonstrated at a SAM-modified electrode where the immobilised DNA serves as an ion gate, opening upon hybridisation as the DNA changes from flexible to rigid.



## CSe<sub>2</sub>-free synthesis of [1,3]diselenole-2-thione and its application to syntheses of iodinated tetraselenafulvalenes (TSeFs)

Tatsuro Imakubo\* and Takashi Shirahata

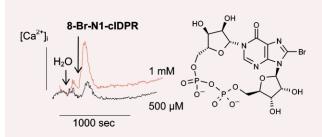
A new CSe<sub>2</sub>-free protocol for the synthesis of [1,3]diselenole-2-thione has been developed. This new method is applicable to gram-scale synthesis and it can easily be converted to TSeF derivatives without isomerization of the [1,3]diselenole ring.



# P-C versus C-H bond cleavage in coordinated bis(dimethylphosphino)methane: controlled access to either phosphinite or functionalized diphosphine complexes

Roberto Quesada, Javier Ruiz,\* Víctor Riera, Santiago García-Granda and M. Rosario Díaz

Basic treatment of coordinated dmpm allows access to either phosphinite or functionalized diphosphine complexes, mediated by selective P–C or C–H bond cleavage.



## First enzymatic synthesis of an N1-cyclised cADPR (cyclic-ADP ribose) analogue with a hypoxanthine partial structure: discovery of a membrane permeant cADPR agonist

Gerd K. Wagner, Steven Black, Andreas H. Guse and Barry V. L. Potter\*

In contrast to its parent nucleotide **NHD**<sup>+</sup>, **8-Br-NHD**<sup>+</sup> is cyclised by the ADP ribosyl cyclase from *A. californica* at the N1, and not at the N7 position. The resulting **8-Br-N**<sup>1</sup>-**cIDPR** acts as a membrane-permeant inducer of Ca<sup>2+</sup> release in human T cells.



Stabilised phosphorus(I) and arsenic(I) iodide: readily-synthesised reagents for low oxidation state main group chemistry

Bobby D. Ellis, Michelle Carlesimo and Charles L. B. Macdonald\*

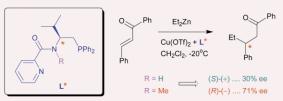


We report a surprisingly facile synthesis of base-stabilised phosphorus(I) and arsenic(I) iodide salts, which are convenient reagents for the synthesis of new low oxidation state main group compounds. The remarkable stability of such compounds is explained by density functional theory calculations.

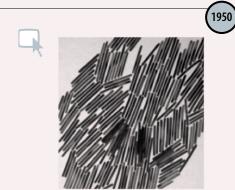


A long-range chiral relay *via* tertiary amide group in asymmetric catalysis: new amino acid-derived N,P-ligands for copper-catalysed conjugate addition

Andrei V. Malkov,\* John B. Hand and Pavel Kočovský\*



The tertiary amide group effectively relays chiral information from the ligand backbone to the reaction centre. It controls the extent and the sense of the chiral induction in Cu-catalysed conjugate addition of diethylzinc to enones with up to 87% ee.



Nanorod shape separation using surfactant assisted self-assembly

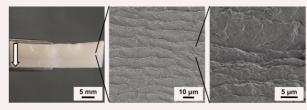
Nikhil R Jana

High quality single size nanorods can be separated from polydisperse samples using surfactant-assisted nanorod self-assembly.



Formation of calcium phosphate having a hierarchically laminated architecture through periodic precipitation in organic gel

Hiroaki Imai,\* Shusei Tatara, Kozue Furuichi and Yuya Oaki

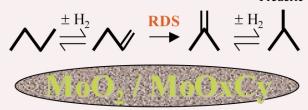


Hierarchically laminated architecture of calcium phosphate was spontaneously produced through Liesegang periodic precipitation in a gel matrix of poly(acrylic acid) containing phosphate anions by diffusion of calcium cations.

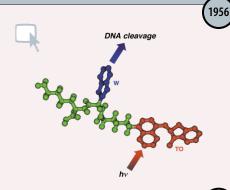


New insights into the reaction mechanism and the rate-determining step of *n*-butane hydroisomerisation over reduced MoO<sub>3</sub> catalysts

Frederic C. Meunier\*



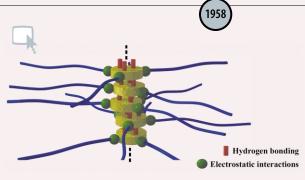
Both the nature of the reaction mechanism and the rate-determining step of n-butane hydroisomerisation over reduced  $MoO_3$  was determined by the comparison of the proportions of the minor products formed with thermodynamic data.



#### Photosensitized DNA cleavage promoted by amino acids

Kerry P. Mahon, Jr., Rodrigo F. Ortiz-Meoz, Erin G. Prestwich and Shana O. Kelley\*

A novel class of DNA cleavage agents are reported that derive activity from amino acids tethered to a photoactive intercalator.



Combination of ionic self-assembly and hydrogen bonding as a tool for the synthesis of liquid-crystalline materials and organogelators from a simple building block

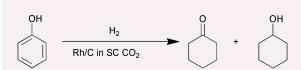
Franck Camerel and Charl F. J. Faul\*

Facile combination of hydrogen bonding and the ionic self-assembly (ISA) process leads to the production of organized materials and fiber-containing organogel superstructures from a functionalised  $C_3$ -symmetrical disk.

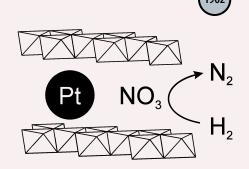


## Catalytic ring hydrogenation of phenol under supercritical carbon dioxide

Chandrashekhar V. Rode, Uday D. Joshi, Osamu Sato and Masayuki Shirai\*



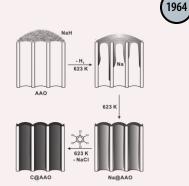
A charcoal-supported rhodium catalyst was highly active for the ring hydrogenation of phenol and cresols under supercritical carbon dioxide.



## Decomposition of Pt-intercalated hydrotalcite-like nanocomposites to produce micro/mesoporous catalysts

Masato Machida\* and Shin Hamada

Platinum intercalated into a hydrotalcite-like solid was found to catalytically reduce interlayer nitrate to  $N_2/N_2O$  so as to give rise to a large surface area micro/mesoporous structure.

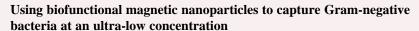


New nanotube synthesis strategy – application of sodium nanotubes formed inside anodic aluminium oxide as a reactive template

Lung-Shen Wang, Chi-Young Lee\* and Hsin-Tien Chiu\*

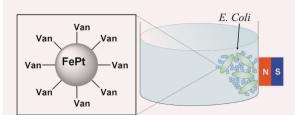
Decomposing NaH thermally on anodic aluminium oxide (AAO) generated Na@AAO, Na nanotubes inside the AAO channels. Na@AAO is employed to react with  $C_6Cl_6$  to form highly aligned bundles of nearly amorphous open-ended carbon nanotubes inside AAO.







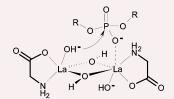
Hongwei Gu, Pak-Leung Ho, Kenneth WT Tsang, Chun-Wing Yu and Bing Xu\*



Elaborated with vancomycin (Van), FePt magnetic nanoparticles (~4 nm) capture E. coli at 15 cfu mL<sup>-1</sup>, suggesting that magnetic nanoparticles may help detect other biological substrates at exceedingly low concentrations.



Unusually high phosphodiesterolytic activity of La(III) hydroxide complexes stabilized by glycine derivatives



Felipe Medrano, Antonio Calderón and Anatoly K. Yatsimirsky\*

Glycine and N,N-dimethylglycine stabilize La(III) hydroxide complexes of the type La<sub>2</sub>L<sub>2</sub>(OH)<sub>4</sub> which possess phosphodiesterolytic activity close to that observed with most active tetravalent cations like Ce(IV).



1966

Yongzhong Bian, Rongming Wang, Jianzhuang Jiang, Chi-Hang Lee, Jinzhi Wang and Dennis K. P. Ng

Synthesis, spectroscopic characterisation and structure of the first chiral heteroleptic bis(phthalocyaninato) rare earth complexes



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

The 'Trinity' helix: synthesis and structural characterisation of a  $C_3$ -symmetric tris-bidentate ligand and its coordination to Ag(I)



Joaquim Crusats, Josep Claret, Ismael Díez-Pérez, Zoubir El-Hachemi, Héctor García-Ortega, Raimon Rubires, Francesc Sagués and Josep M. Ribó

Chiral shape and enantioselective growth of colloidal particles of selfassembled meso-tetra(phenyl and 4-sulfonatophenyl)porphyrins

Steven D. Bull. Matthew G. Davidson, Andrew L. Johnson, Diane E. J. E. Robinson and Mary F. Mahon

Synthesis, structure and catalytic activity of an air-stable titanium triflate, supported by an amine tris(phenolate) ligand



Dates, venues and contact details of forthcoming events.

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