

**Cover**  
Self-adaptability and conformations in oxothiomolybdate rings.

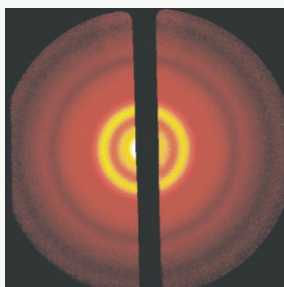


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[www.rsc.org/chembiol](http://www.rsc.org/chembiol)

# contents

## FOCUS ARTICLE

2185



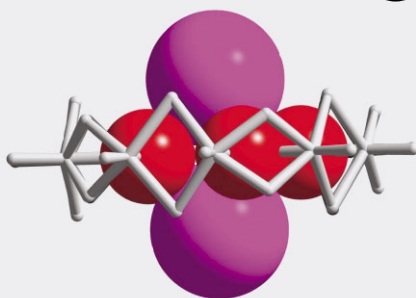
### Chemistry and biology in the new age

Nina Hall

Looking back at the visionary work of Ahmed Zewail in probing the motion of atoms at the femtosecond level, and looking forward to his work in the realm of molecular complexity, using ultrafast electron diffraction to 'watch' reactions.

## FEATURE ARTICLE

2189



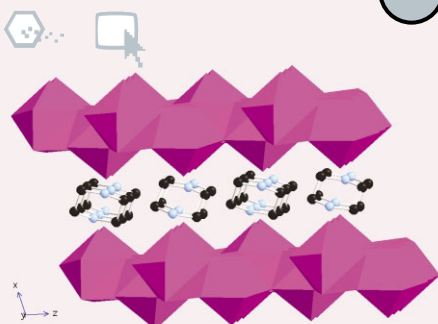
### Cyclic molecular materials based on $[M_2O_2S_2]^{2+}$ cores (M = Mo or W)

Emmanuel Cadot and Francis Sécheresse

The self condensation of the  $[M_2O_2S_2]^{2+}$  oxothio-fragment produces an extended family of cyclic molecular materials. The resulting cycles, differing by their size and shape, exhibit a cationic open cavity, which can interact with small anionic molecules, such as phosphates, dicarboxylates and metalates. The cyclic architecture is flexible and self-adaptable to the nature of the encapsulated substrate.

## COMMUNICATIONS

2198



### The first organically templated thorium compounds, $[C_4N_2H_{12}]_{0.5}[ThF_5]$ and $[C_5N_2H_{14}][ThF_6] \cdot 0.5H_2O$

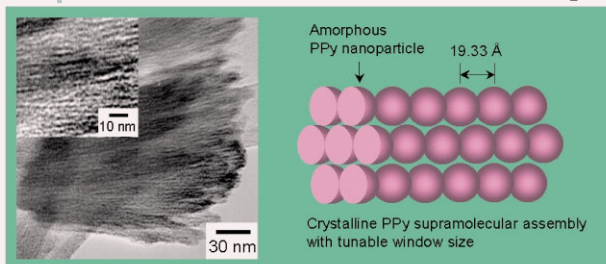
Jong-Young Kim, Alexander J. Norquist and Dermot O'Hare\*

Organically templated thorium compounds were synthesized for the first time under hydrothermal conditions using amine templates; the piperazine containing compound consists of 2-D layers, while the 2-methylpiperazine phase contains unprecedented 1-D chains of face-sharing  $ThF_9$  polyhedra.

2200

### Novel crystalline supramolecular assemblies of amorphous polypyrrole nanoparticles through surfactant templating

Jyongsik Jang\* and Joon Hak Oh

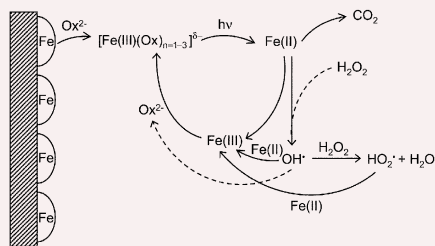


A lamellar-structured crystalline polypyrrole (PPy) supramolecular assembly was prepared by surfactant templating. Regularly linked amorphous PPy nanoparticles with tunable window sizes could play the role of crystalline lattices in the supramolecular assembly.

2202

### Abatement of oxalates catalyzed by Fe-silica structured surfaces *via* cyclic carboxylate intermediates in photo-Fenton reactions

Anna Bozzi, Tatiana Yuranova, J. Mielczarski, A. Lopez and J. Kiwi



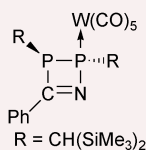
Novel silica/Fe structured fabrics were observed to degrade oxalates only under light irradiation showing the formation and disappearance of Fe-carboxylates and the concomitant recycling of the resulting Fe-ions back to the structured catalyst surface.

2204

### Synthesis of the first 2,3-dihydro-1,2,3-azadiphosphete complex

E. Ionescu, P. G. Jones and R. Streubel\*

Synthesis of the first 2,3-dihydro-1,2,3-azadiphosphete complex was achieved by heating a solution of a 2*H*-azaphosphirene complex.

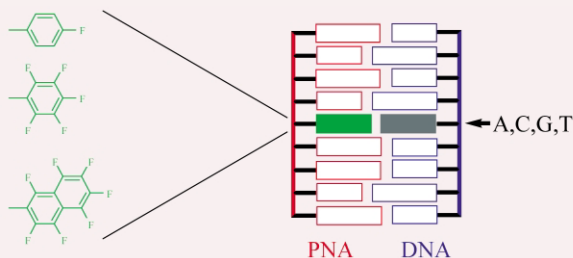


2206

### Fluoroaromatic universal bases in peptide nucleic acids

Kathryn A. Frey and Stephen A. Woski\*

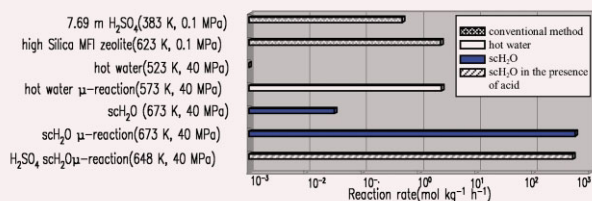
Fluoroaromatic peptide nucleic acid residues were found to possess little base discrimination when incorporated into PNA-DNA double helices.



2208

### Innovation in a chemical reaction process using a supercritical water microreaction system: environmentally friendly production of $\epsilon$ -caprolactam

Yutaka Ikushima,\* Kiyotaka Hatakeda, Masahiro Sato, Osamu Sato and Masahiko Arai

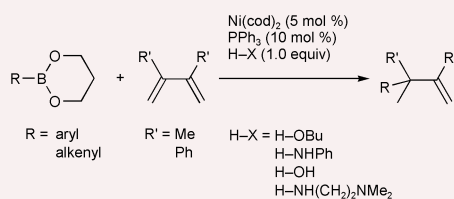


The sch<sub>2</sub>O  $\mu$ -reaction system achieves nearly 100% selectivity in satisfactory yield for  $\epsilon$ -caprolactam production.

2210

**Nickel-catalysed addition of organoboronates to 1,3-dienes**

Eiji Shirakawa,\* Go Takahashi, Teruhisa Tsuchimoto and Yusuke Kawakami

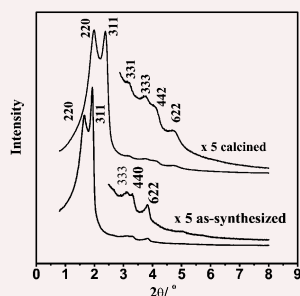


The hydroarylation and -alkenylation of 1,3-dienes were accomplished by activation of carbon–boron bonds with a nickel–triphenylphosphine catalyst in combination with a stoichiometric amount of water, an alcohol or an amine as a proton donor.

2212

**A novel ordered cubic mesoporous silica templated with tri-head group quaternary ammonium surfactant**

Shaodian Shen, Yuqi Li, Zhendong Zhang, Jie Fan, Bo Tu, Wuzong Zhou\* and Dongyuan Zhao\*

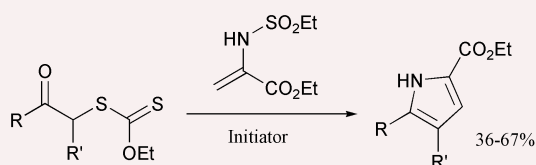


A novel ordered cubic silica mesostructure (space group  $Fd\bar{3}m$ ) has been synthesized with tri-head group quaternary ammonium surfactants  $C_{m-2-3-1}$  ( $m = 14, 16, 18$ ) under basic conditions at low temperature.

2214

**A new synthesis of pyrroles**

Béatrice Quiclet-Sire, Frédérique Wendeborn (née Bertrand) and Samir Z. Zard\*

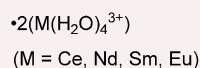
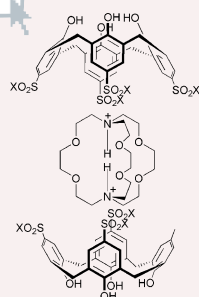


The radical reaction between an *N*-ethylsulfonylenamide and an  $\alpha$ -xanthyl ketone gives an intermediate  $\gamma$ -keto imine which spontaneously ring-closes to the pyrrole.

2216

**Capture of di-protonated [2.2.2]cryptand in the cavity of two *p*-sulfonated calixarenes as part of 2-D bi-layer lanthanide coordination polymers**

Scott J. Dalgarno and Colin L. Raston\*

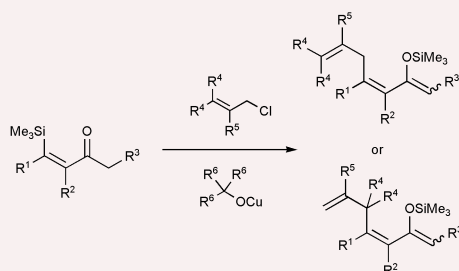


2-D bi-layer coordination polymers based on Ce, Nd, Sm and Eu(III) complexes of *p*-sulfonatocalix[4]arene), with axially elongated diprotonated [2.2.2]cryptand in the cavity of two *p*-sulfonatocalix[4]arenes are described.

2218

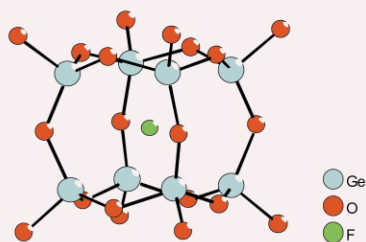
**First anionic silyl migration from  $sp^2$  carbon to carbonyl oxygen. Stereospecific allylation of (*Z*)- $\beta$ -trimethylsilyl- $\alpha,\beta$ -unsaturated ketones**

Haruhiko Taguchi, Hiroko Miyashita, Akira Tsubouchi and Takeshi Takeda\*



(*Z*)- $\beta$ -Trimethylsilyl- $\alpha,\beta$ -unsaturated ketones were treated with copper(I) alkoxides and allylic halides to afford enol silyl ethers of  $\beta$ -alk-2-enyl- $\alpha,\beta$ -unsaturated ketones with complete retention of configuration.

2220



### Synthesis and structure of fluoride-containing GeO<sub>2</sub> analogues of zeolite double four-ring building units

Luis A. Villaescusa, Philip Lightfoot\* and Russell E. Morris\*

The first examples of isolated double four-membered ring (D4R) anionic building units containing germanium have been synthesised hydrothermally and their structures solved from powder X-ray diffraction.

2222

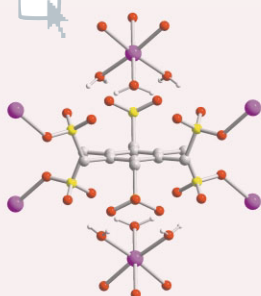


### C–Cl/Si–H Exchange catalysed by P,N-chelated Pt(II) complexes

Frank Stöhr, Dietmar Sturmayer and Ulrich Schubert\*

Alkyl chlorides R–Cl are dehalogenated by HSiMe<sub>2</sub>Ph in the presence of a catalytic amount of the complex [(κ<sup>2</sup>-P,N)-Ph<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>NMe<sub>2</sub>]PtMeCl to give R–H and ClSiMe<sub>2</sub>Ph. This is the first example of a hydrodechlorination reaction by hydrosilanes.

2224

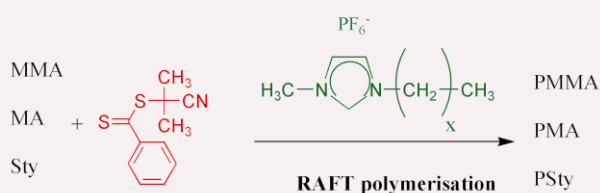


### An open channel coordination framework sustained by cooperative primary and secondary sphere interactions

Sean A. Dalrymple and George K. H. Shimizu\*

An Al<sup>3+</sup> complex of a novel hexasulfonate ligand is reported where each metal center is stabilized by both primary and secondary sphere coordination to form a 3-D channel structure.

2226

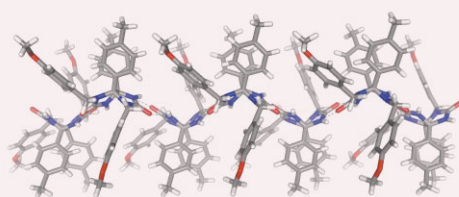


### First report of reversible addition–fragmentation chain transfer (RAFT) polymerisation in room temperature ionic liquids

Sébastien Perrier, Thomas P. Davis,\* Adrian J. Carmichael and David M. Haddleton

Efficient living radical polymerisation *via* the reversible addition–fragmentation chain transfer (RAFT) process is reported for the first time in ionic liquids for styrene, methyl methacrylate and methyl acrylate monomers.

2228



### New supramolecular organization for a glycoluril: chiral hydrogen-bonded ribbons

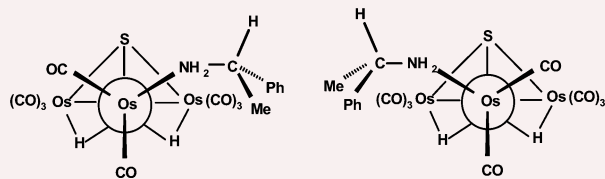
Darren W. Johnson, Liam C. Palmer, Fraser Hof, Peter M. Iovine and Julius Rebek Jr.\*

Severe twisting of the bridgehead phenyl rings in a series of glycolurils produces chiral hydrogen-bonded ribbons in the crystalline state. Complementary DFT and NMR studies analyze the nature of this twist.

2230

### Slow epimerization of stereochemically rigid diastereomers of the equatorially substituted cluster $[\text{Os}_3\text{H}_2(\mu_3\text{-S})(\text{CO})_8\{(\text{S})\text{-PhCHMeNH}_2\}]$

Antony J. Deeming,\* Caroline S. Forth, Graeme Hogarth, David Markham, Jade O. Prince and Jonathan W. Steed

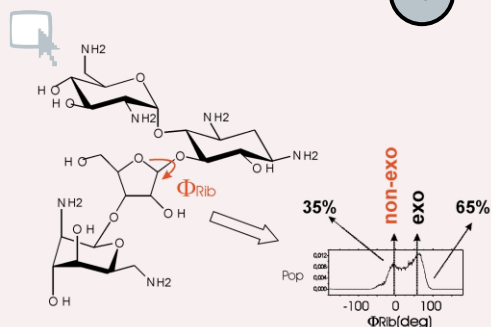


Turnstile rotation is suppressed in the equatorially substituted cluster  $[\text{Os}_3(\mu\text{-H})_2(\mu_3\text{-S})(\text{CO})_8\{(\text{S})\text{-PhCHMeNH}_2\}]$ . Remarkably the two diastereomers shown do not interconvert at room temperature and epimerize only slowly at 90 °C.

2232

### Experimental evidence for the existence of non-*exo*-anomeric conformations in branched oligosaccharides: the neomycin-B case

Juan Luis Asensio,\* Ana Hidalgo, Igor Cuesta, Carlos Gonzalez, Javier Cañada, Cristina Vicent, Jose Luis Chiara, Gabriel Cuevas and Jesus Jimenez-Barbero

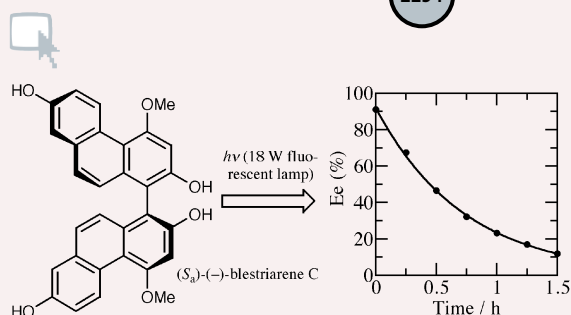


In neomycin B, a branched oligosaccharide antibiotic, for the first time in natural sugars, a large amount of non-*exo*-anomeric conformation is experimentally detected, in solution. Contacts between non-vicinal sugar units lead to an enhanced flexibility of the ribose glycosidic torsion  $\phi$ .

2234

### First determination of the absolute stereochemistry of a naturally occurring 1,1'-biphenanthrene, (–)-blestriarene C, and its unexpected photoracemization

Tetsutaro Hattori,\* Yuhi Shimazumi, Osamu Yamabe, Eiji Koshiishi and Sotaro Miyano\*

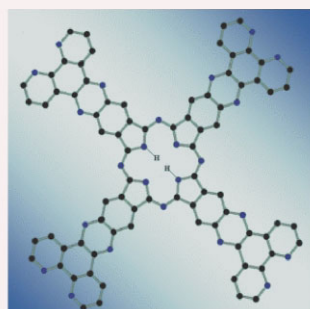


A naturally occurring 1,1'-biphenanthrene, blestriarene C, was prepared and its absolute stereochemistry was determined to be  $S_a(-)$  by an empirical method, during which the compound was found to undergo rapid photoracemization even under ambient light exposure.

2236

### A novel fully conjugated phenanthroline-appended phthalocyanine: synthesis and characterisation

Julia Rusanova, Melanie Pilkington\* and Silvio Decurtins

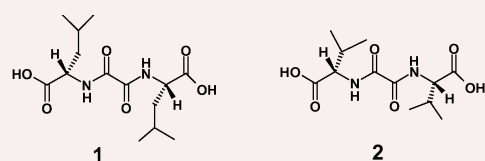


The synthesis and characterisation of a novel fully conjugated, tetrasubstituted  $\text{H}_2\text{Pc}$ , containing four peripheral diimine binding sites, fused *via* pyrazine bridges to a metal free phthalocyanine core is described.

2238

### Gels with exceptional thermal stability formed by bis(amino acid) oxalamide gelators and solvents of low polarity

Janja Makarević, Milan Jokić, Leo Frkanec, Darinka Katalenić and Mladen Žinić\*

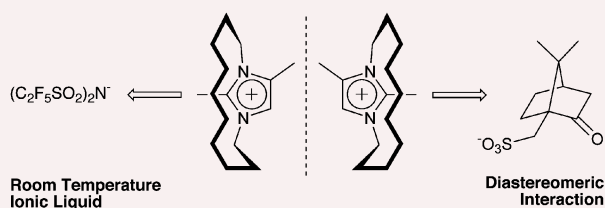


The oxalamide gelators **1** and **2** form common thermo-reversible gels and also gels of exceptional thermal stability which can be heated up to 50 °C higher temperatures than the bp of the gelled solvent.

2240

**Design and synthesis of a novel imidazolium-based ionic liquid with planar chirality**

Yasuhiro Ishida, Hiroyuki Miyauchi and Kazuhiko Saigo\*

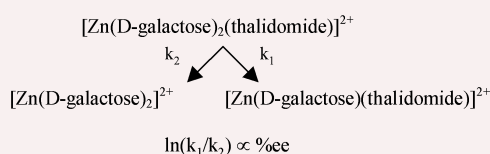


As the first example of a planar chiral ionic liquid, a cyclophane-type imidazolium salt was synthesized; its potential use in chiral recognition was demonstrated by  $^1\text{H}$  NMR studies.

2242

**Quantitative determination of the enantiomeric composition of thalidomide solutions by electrospray ionization tandem mass spectrometry**

Daniella V. Augusti, Rodinei Augusti, Fernando Carazza and R. Graham Cooks\*

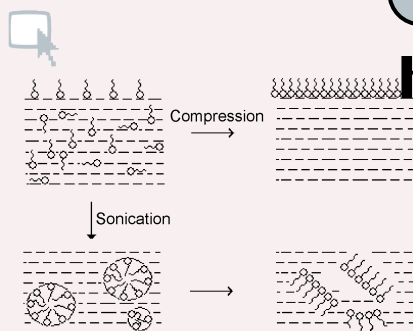


A fast method for quantitative chiral analysis of thalidomide solutions using MS/MS is described.

2244

**Sonication induced sheet formation at the air–water interface**

K. S. Satheeshkumar and R. Jayakumar\*

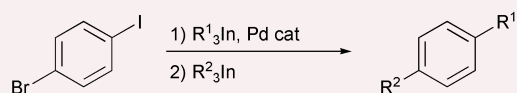
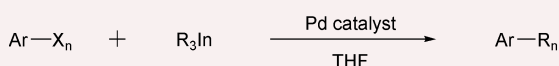


Enhanced  $\beta$ -sheet content of prion fragment PrP (113-127) is observed when compressed at the air–water interface and also by sonication, indicating that molecular crowding at the interface plays a crucial role in assembly formation.

2246

**Multifold and sequential cross-coupling reactions with indium organometallics**

Miguel A. Pena, Ignacio Pérez, José Pérez Sestelo\* and Luis A. Sarandeses\*



Multifold and sequential palladium-catalyzed cross-coupling reactions can be performed with triorganoindium compounds using only a small excess of the organometallic reagent, low catalyst charge loading and short reaction times.

2248

**A selective chromogenic reagent for cyanide determination**

Jose V. Ros-Lis, Ramón Martínez-Máñez\* and Juan Soto



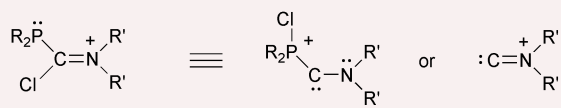
A chromogenic reagent for cyanide determination in water based on the reaction of this anion with a squaraine derivative functionalized with ether chains has been developed.

2250



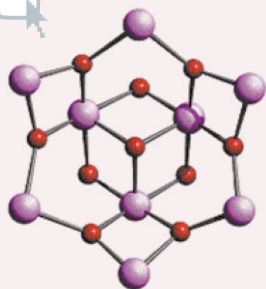
### C-Phosphanil-C-chloroiminium salts as electrophilic carbene synthetic equivalents

Nathalie Merceron, Antoine Baceiredo, Heinz Gornitzka and Guy Bertrand\*



C-Phosphanil-C-chloroiminium salts formally react as phosphonio(amino)carbenes with *tert*-butyl isocyanide and trimethylphosphine, and as  $\text{R}_2\text{NC}^+$  with vinyl ether and diisopropylamine.

2252

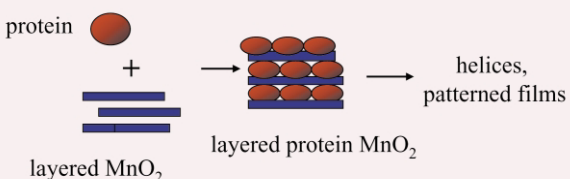


### A new class of single-molecule magnet: $[\text{Mn}_9\text{O}_7(\text{OAc})_{11}(\text{thme})(\text{py})_3(\text{H}_2\text{O})_2]$ with an $S = 17/2$ ground state

Euan K. Brechin,\* Monica Soler, James Davidson, David N. Hendrickson, Simon Parsons and George Christou\*

The synthesis, structure and magnetic properties of a new mixed-valent manganese cluster are reported. The complex displays strong out-of-phase signals in ac susceptibility studies that establish it as a single-molecule magnet.

2254

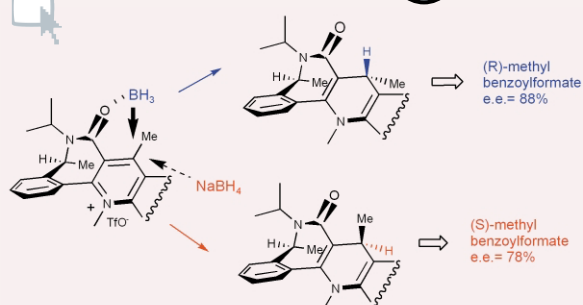


### Colloids, helices, and patterned films made from heme proteins and manganese oxide

Qiuming Gao, Steven L. Suib\* and James F. Rusling\*

Layered manganese oxide colloids and proteins were combined into new materials with enzyme-like peroxidase activity with the proteins intercalated between layers. Further processing gave macroscopic helical structures and patterned films.

2256

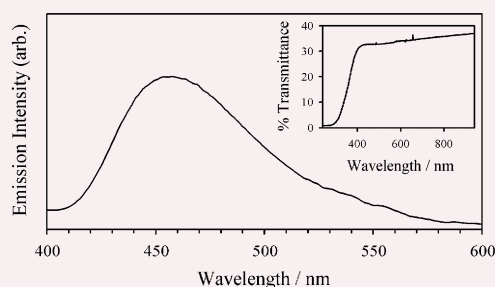


### Atropisomeric quinolinium salt promoting the access to both enantiomeric forms of methyl mandelate: a versatile NADH mimic

Jean-Luc Vasse, Vincent Levacher,\* Jean Bourguignon and Georges Dupas

This communication reports the reduction of methyl benzoylformate with a new class of NADH mimics, affording either (*R*)-methyl mandelate in up to 88% ee or (*S*)-methyl mandelate in 78% ee starting from the same  $\text{NAD}^+$  mimic salt.

2258

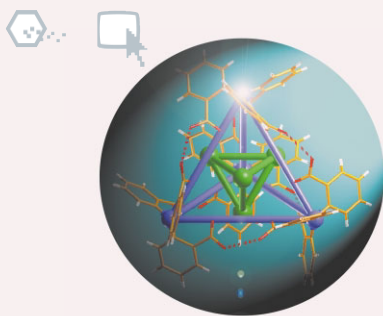


### Photoluminescent carbon nitride films grown by vapor transport of carbon nitride powders

Jianjun Wang, Dale R. Miller and Edward G. Gillan\*

Nitrogen-rich carbon nitrides ( $\text{C}_3\text{N}_{4+x}$ ) evaporate above 600 °C and recondense as pale yellow films near 300 °C. The films strongly absorb in the near UV and photoluminesce in the blue region.

2260



### Spontaneous assembly of a hydrogen-bonded tetrahedron

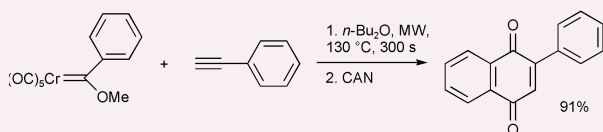
Jason E. Field, Marianny Y. Combariza, Richard W. Vachet and D. Venkataraman\*

Triphenylamine *ortho*-tricarboxylic acid has been synthesized and the crystal structure reported. This molecule is shown to spontaneously self-assemble into a hydrogen-bonded tetrahedron. Furthermore, Electrospray Ionization Mass Spectroscopy shows evidence for the stability of such aggregates from an ethanol/water solution.

2262

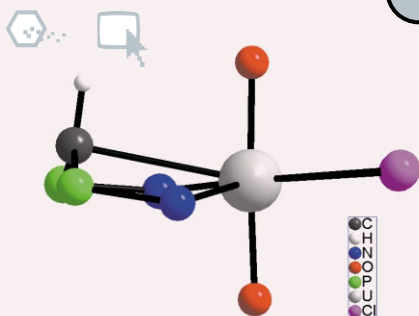
### The microwave-assisted Dötz benzannulation process

Edward J. Hutchinson, William J. Kerr\* and Euan J. Magennis



The Dötz reaction has been shown to proceed remarkably rapidly and with enhanced efficiency under developed microwave-assisted conditions and gives benzannulation products in up to excellent yields following only 5 min reaction time.

2264

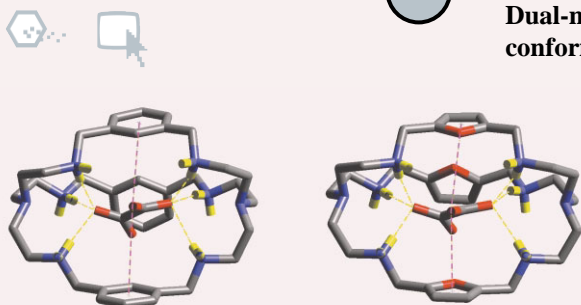


### The first uranyl–methine carbon bond; a complex with out-of-plane uranyl equatorial coordination

Mark J. Sarsfield,\* Madeleine Helliwell and David Collison

Treatment of  $[\text{UO}_2\text{Cl}_2(\text{thf})_3]$  in thf with one equivalent of  $[\text{Na}\{\text{CH}(\text{Ph}_2\text{P}=\text{NSiMe}_3)_2\}]$  yields an unusual uranyl chloro-bridged dimer containing a uranium(VI)–carbon bond as part of a tridentate bis(iminophosphorano)methanide chelate complex. The methine carbon is significantly displaced from the uranyl equatorial plane.

2266

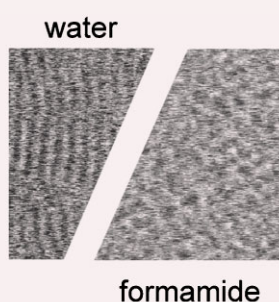


### Dual-mode recognition of oxalate by protonated azacryptate hosts; conformational response of the guest maximizes $\pi$ -stacking interactions

Jane Nelson,\* Mark Nieuwenhuyzen, Ibolya Pál and Raewyn M. Town

Dual mode  $\text{NH}\cdots\text{O}_{(\text{ox})}$  and  $\text{C}=\text{O}$  to  $\pi$  recognition leads to exceptionally high stability constants for oxalate complexation by *m*-xylyl or 2,5-furano spaced cryptates.

2268



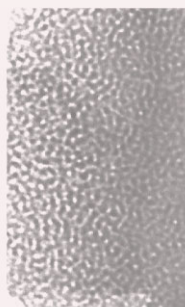
### Shape of tetradecyltrimethylammonium chloride aggregates at liquid/solid interfaces in mixtures of water and formamide

Franck P. Duval and Gregory G. Warr\*

Tetradecyltrimethylammonium chloride forms an adsorbed layer of micelle-like aggregates on mica in water/formamide mixtures at all compositions. As the formamide content is increased, the fully-developed adsorbed layer is formed at higher surfactant concentrations and the adsorbed aggregates are transformed from cylinders into globules.



2270

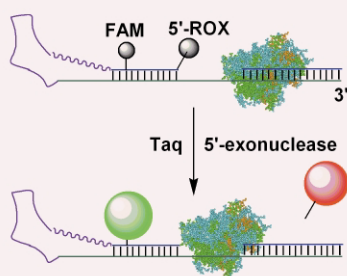


### Effect of solution chemistry and speciation on shelf-life of silica sols and characteristics of deposited mesoporous thin films

J. C. Birnbaum,\* X. Li, C. R. Yonker, G. E. Fryxell and S. Baskaran

This communication describes a thorough investigation of the effect of solution chemistry on the shelf-life of silica sols. Important results are presented that are of interest in the field of low  $k$  materials. The temperature and time dependency of the oligomeric silicate species in the sol provides novel insight for the scientific community involved in mesoporous material synthesis.

2272



### Intramolecular TaqMan probes for genetic analysis

Antonio Solinas, Nicola Thelwell and Tom Brown\*

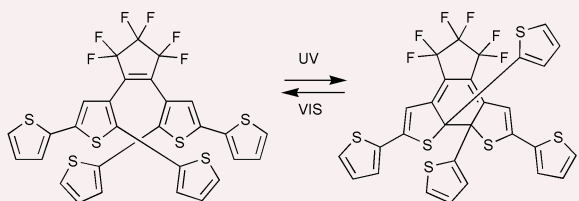
We describe a new intramolecular version of the TaqMan assay which is used widely in the analysis of single nucleotide polymorphisms. The new method offers advantages over existing technologies.

2274



### Regulating $\pi$ -conjugated pathways using a photochromic 1,2-dithienylcyclopentene

Andrea Peters, Robert McDonald and Neil R. Branda\*

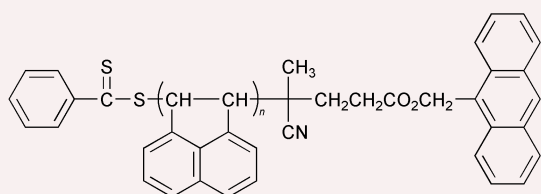


Linear  $\pi$ -conjugation is reversibly re-routed by irradiation of a photochromic bis(terthiophene).

2276

### Synthesis of light harvesting polymers by RAFT methods

Ming Chen, Kenneth P. Ghiggino,\* Albert W. H. Mau, Ezio Rizzardo, San H. Thang\* and Gerard J. Wilson



Polymers prepared by RAFT polymerisation methods containing acenaphthyl energy donors and a terminal anthryl energy acceptor have a narrow molecular weight distribution and exhibit excitation energy transfer efficiencies up to 70%.

## ADDITIONS AND CORRECTIONS

2278

Gopal Das, Parimal K. Bharadwaj,  
Debjani Ghosh, Beauty Chaudhuri  
and Rupendranath Bannerjee

**Synthesis and structure of the  $[\text{Mn}^{\text{IV}}(\text{biguanide})_3]^{4+}$  ion: the simplest source for water-stable manganese(IV)**

2278

Peter B. Hitchcock, John F. Nixon  
and Nurgün Sakarya

**Syntheses of the novel cage compounds  $\text{P}_3\text{Se}_3\text{C}_3\text{Bu}^t_3$  and  $\text{P}_3\text{Se}_4\text{C}_3\text{Bu}^t_3$  and an unusual insertion reaction of  $[\text{PtCl}_2(\text{PMe}_3)]$  into the Se–Se bond of the latter to give the six-coordinate Pt(IV) complex  $[\text{PtCl}_2(\text{PMe}_3)\text{P}_3\text{Se}_4\text{C}_3\text{Bu}^t_3]$**

2278

Peter B. Hitchcock, John F. Nixon  
and Nurgün Sakarya Büyükkidan

**Remarkable organophosphorus cage compounds from the reaction of cobaltocene and the triphosphole  $\text{P}_3\text{C}_2\text{Bu}^t_2\text{CH}(\text{SiMe}_3)_2$ : crystal and molecular structures of  $[\text{Co}(\eta^5\text{-C}_5\text{H}_5)(\eta^4\text{-C}_4\text{H}_4\text{CHCHP}_6\text{C}_4\text{Bu}^t_4)]$  and  $\text{P}_6\text{C}_4\text{Bu}^t_4\text{CH}(\text{SiMe}_3)$**

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\* Indicates the author for correspondence: see article for contact details.  
Supplementary crystallographic data are available: see article for further information.



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