



Cover
Crambe Crambe, a marine sponge from which crambescidin alkaloids have been isolated (pp. 253–265). Photo courtesy of Miquel Pontes (<http://marenostrum.org>)



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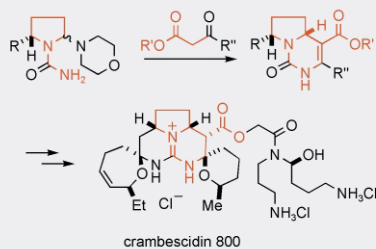
contents

FEATURE ARTICLE

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The tethered Biginelli condensation in natural product synthesis

Zachary D. Aron and Larry E. Overman*



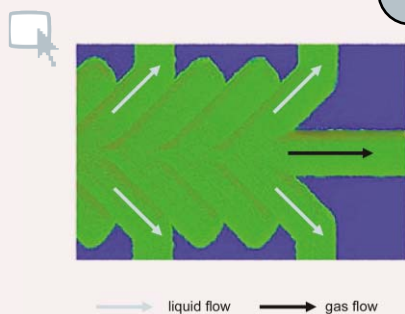
This review describes the development of the tethered Biginelli condensation and its application to the total synthesis of structurally complex, bioactive guanidine alkaloids.

COMMUNICATIONS

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Continuous laminar evaporation: micron-scale distillation

Robert C. R. Wootton and Andrew J. deMello*

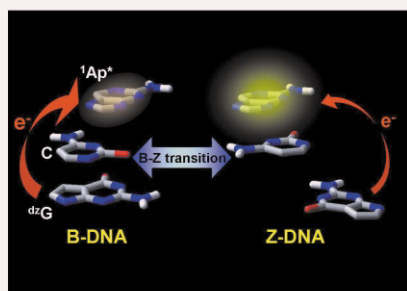


Micron scale distillation within continuous flow microfluidic systems is achieved using laminar flow regimes and carrier gases to control liquid movement.

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Fluorescence properties of 2-aminopurine–cytidine–7-deazaguanine (5'-ApC^{dz}G-3') trimer in B- and Z-DNA

Takumi Kimura, Kiyohiko Kawai and Tetsuro Majima*



To test the possibility of using 2-aminopurine as a Z-DNA probe, the effect of the B–Z transition on electron transfer quenching of Ap by guanine and 7-deazaguanine was investigated.

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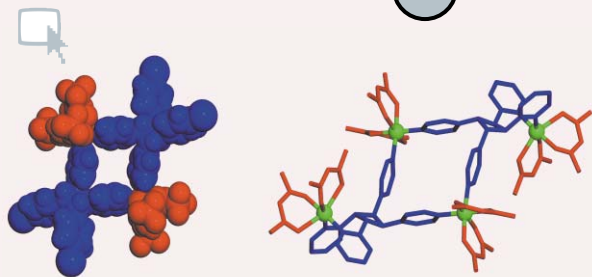
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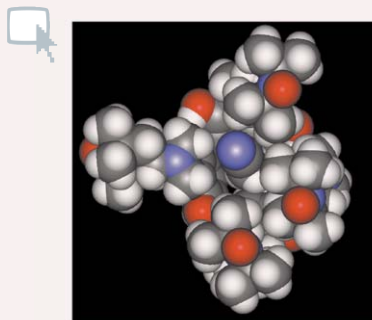


Self-assembled metal–organic squares derived from linear templates as exemplified by a polydentate ligand that provides access to both a polygon and polyhedron

Giannis S. Papaefstathiou, Tamara D. Hamilton, Tomislav Friščić and Leonard R. MacGillivray*

Bi- and polyfunctional pyridyl-cyclobutanes obtained from template-directed syntheses conducted in the solid state self-assemble with the Cu(II) ion to form di- and tetranuclear metal–organic squares.

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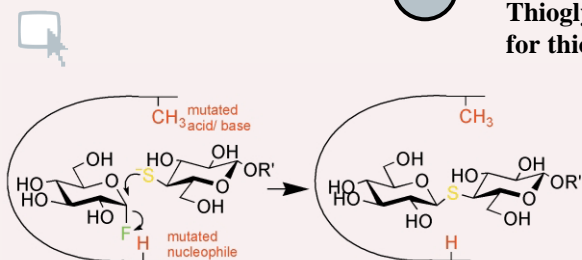


Spin labeling monitors weak host–guest interactions

Lenz Kröck, Alexander Shivanyuk, David B. Goodin and Julius Rebek, Jr.*

Rapid host–guest interactions can be detected by EPR using spin-labeled cavitands.

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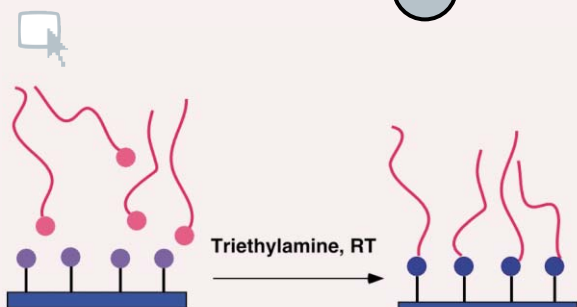


Thioglycosynthases: double mutant glycosidases that serve as scaffolds for thio glycoside synthesis

Michael Jahn, Hongming Chen, Johannes Müllegger, Jennifer Marles, R. Antony J. Warren and Stephen G. Withers

A double mutant, retaining glycosidase that lacks both the catalytic nucleophile and the catalytic acid/base residues efficiently catalyzes thio glycoside formation from a glycosyl fluoride donor and thiosugar acceptors.

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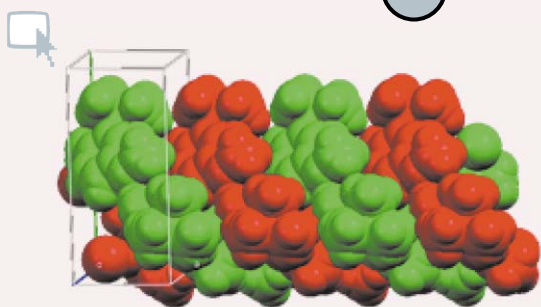


Room-temperature one-step immobilization of rod-like helical polymer onto hydrophilic substrates

Guangqing Guo, Masanobu Naito, Michiya Fujiki,* Anubhav Saxena, Kento Okoshi, Yonggang Yang, Masaaki Ishikawa and Takahiro Hagihara

A facile, one-pot immobilization method for a rigid rod-like helical polysilane, poly(*n*-decyl-*i*-butylsilane), was developed onto hydrophilic surfaces at room temperature in the presence of triethylamine as a catalyst.

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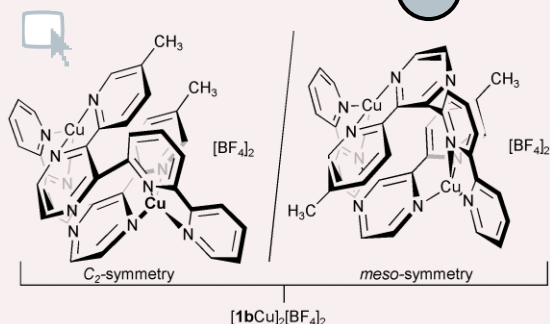


Polymorphism in butylated hydroxy anisole (BHA)

Jennifer A. McMahon, Michael J. Zaworotko and Julius F. Remenar*

Butylated hydroxy anisole (3-BHA) exists as two polymorphs with dramatically different crystal packings sustained by OH⋯ether supramolecular heterosynthons: double helical chains (Form I, alongside) and a discrete hexameric assembly (Form II).

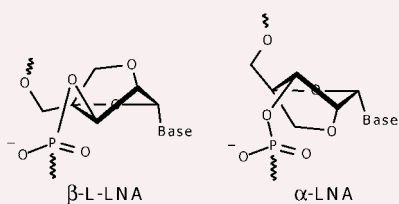
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**In flagrante metallo-cyclophane self-assembly?**

Peter J. Cragg, Fenton R. Heitzler,* Mark J. Howard, Ivan Prokes and Thomas Weyhermüller

The dimeric self-assembly of an alkyl-substituted pyrazine–pyridine hybrid ligand with copper(I) initially affords its sterically congested, C_2 -symmetric stereoisomer, which then undergoes partial isomerisation to a dynamic mixture containing the less crowded *meso*-configured diastereomer.

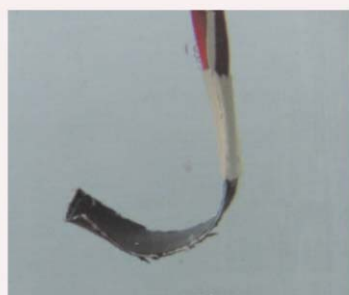
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**Parallel nucleic acid recognition by the LNA (locked nucleic acid) stereoisomers β -L-LNA and α -D-LNA; studies in the mirror image world**

Nanna K. Christensen, Torsten Bryld, Mads D. Sørensen, Khalil Arar, Jesper Wengel and Poul Nielsen*

Two stereoisomers of LNA are evaluated in the mirror-image world by the study of LNA and α -L-LNA and their L-DNA and L-RNA complements. Both β -L-LNA and α -D-LNA display high-affinity parallel stranded RNA-recognition.

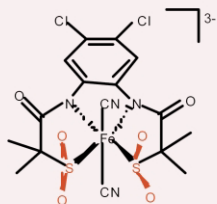
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**Artificial muscle: movement and position control**

T. F. Otero* and M. T. Cortes

All polymeric artificial muscle shows bending under absolute control of the applied current. The position of this device can be controlled by the consumed charge, the speed and movement direction by the magnitude and direction of the current. This device does not show relaxation when the electrical energy is switched off.

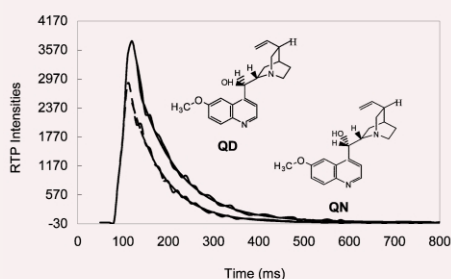
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**Oxygenation of thiolates to *S*-bonded sulfinate in an iron(III) complex related to nitrile hydratase**

Erwan Galardon, Michel Giorgi and Isabelle Artaud*

A new mimic of iron nitrile hydratase containing two sulfinato groups *trans* to two carboxamido nitrogens has been prepared by air oxidation of the corresponding dithiolato complex $[\text{Fe}(\text{N}_2\text{S}_2)\text{Cl}]^{2-}$ in the presence of CN^- .

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**Alteration of room temperature phosphorescence lifetimes of quinine and quinidine by chiral additives**

Yanli Wei, Wing-Hong Chan, Albert W. M. Lee and Carmen W. Huie*

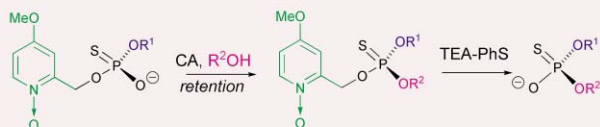
The observation of room temperature phosphorescence lifetime differences between quinine (QN) and quinidine (QD) and the ability to alter these differences based on the addition of chiral counter-ions or surfactants are reported.

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A new approach to stereospecific synthesis of P-chiral phosphorothioates. Preparation of diastereomeric dithymidyl-(3'-5') phosphorothioates

Helena Almer, Tomas Szabo and Jacek Stawinski*



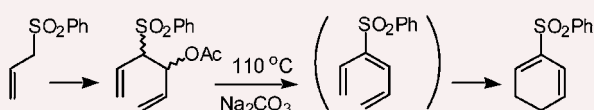
A new method for stereospecific synthesis of P-chiral phosphorothioates based on intramolecular nucleophile catalysis.

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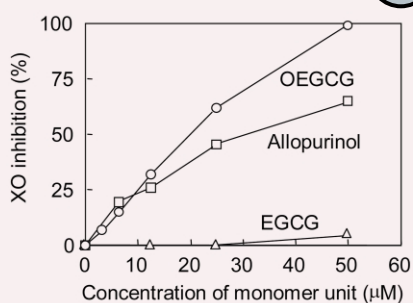
A versatile and concise route to carbocycles using a 1,6-electrocyclic reaction

Svante Brandänge* and Hans Leijonmarck



A 1,6-electrocyclic reaction has been used for the two-step synthesis of substituted 2-phenylsulfonyl-1,3-cyclohexadienes, starting from an α,β -unsaturated aldehyde and an allylic sulfone.

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Oxidative coupling of epigallocatechin gallate amplifies antioxidant activity and inhibits xanthine oxidase activity

Motoichi Kurisawa, Joo Eun Chung, Hiroshi Uyama* and Shiro Kobayashi*

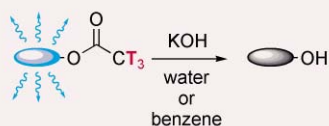
An enzymatically synthesized oligomer (OEGCG) of epigallocatechin gallate showed much greater superoxide scavenging and xanthine oxidase (XO) inhibitory activity than EGCG monomer.

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Scintillation proximity assays for the real-time detection and quantification of the progress of reactions upon solid supports

Mark C. McCairn and Andrew J. Sutherland*



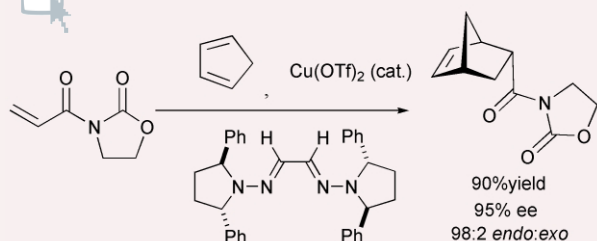
A scintillation proximity assay has been used to detect and quantify, in real-time, the hydrolysis of [^3H]acetate ester from scintillant-containing styrene and PEG-based polymer supports in both benzene and water.

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Glyoxal bis-hydrazones: a new family of nitrogen ligands for asymmetric catalysis

José M. Lassaletta,* Manuel Alcarazo and Rosario Fernández



The introduction of C_2 -symmetric dialkylamino substructures in chiral non-racemic glyoxal bis-hydrazones appears as the key design element for this novel ligand class, as shown in the highly enantioselective copper(II)-catalyzed Diels–Alder reaction.

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Microwave-accelerated synthesis of lengthy and defect-free poly(*m*-phenyleneethynylene)s via AB' and A₂ + BB' polycondensation routes

Anzar Khan and Stefan Hecht*



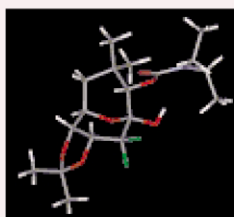
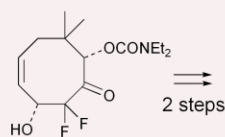
Lengthy and defect-free poly(*m*-phenyleneethynylene)s can be prepared via a novel polycondensation protocol involving an *in-situ* activation/coupling scheme in combination with microwave-acceleration.

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Towards conformationally-locked difluorosugar analogues: an unexpected sense of dihydroxylation

John Fawcett, Gerry A. Griffiths, Jonathan M. Percy,* Stéphane Pintat, Clive A. Smith, Neil S. Spencer and Emi Uneyama

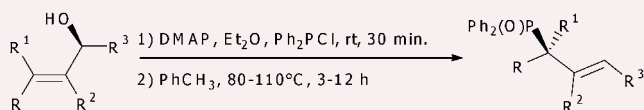


Stereoselective dihydroxylation of a difluorinated cyclooctenone leads to a stable bicyclic hemiacetal via transannular collapse. Molecular topology plays an intriguing role in determining the outcome of this transformation, and related ones.

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Stereoselective [2,3] sigmatropic rearrangement of acyclic allylic phosphinites

Frédéric Liron and Paul Knochel*



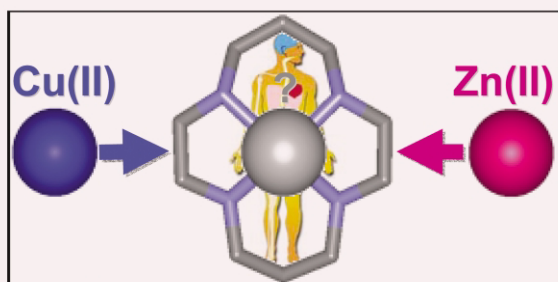
The [2,3] sigmatropic rearrangement of acyclic allylic phosphinites was found to proceed with high stereo- and enantioselectivity, allowing the preparation of chiral allylic phosphine oxides and aminophosphine oxides.

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Anti-viral cyclam macrocycles : rapid zinc uptake at physiological pH

Stephen J. Paisey and Peter J. Sadler*



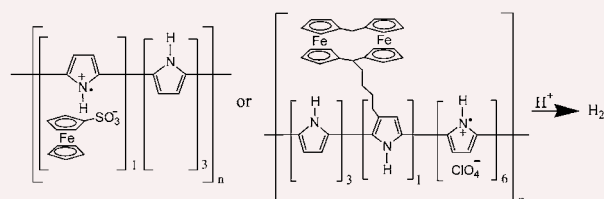
Zn(II) binds to cyclam rapidly at micromolar concentrations and physiological pH, and retards uptake of Cu(II).

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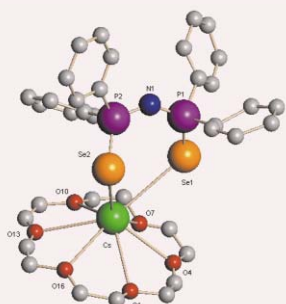
A readily-prepared electrocatalytic coating that is more active than platinum for hydrogen generation in 1 M strong acid

Jun Chen, Junhua Huang, Gerhard F. Swiegers,* Chee O. Too and Gordon G. Wallace*



Coating of a platinum electrode with conducting polypyrrole containing ferrocene sulfonate as counter-ion induces a 0.27 V anodic shift for hydrogen gas evolution in 1 M strong acids and a 7-fold amplification in hydrogen production when poised at -0.44 V.

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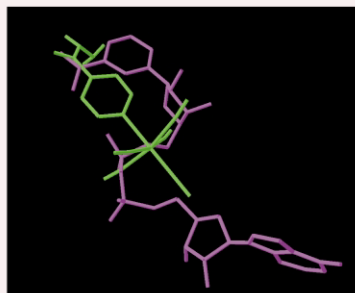


A powerful novel strategy for the preparation of discrete inorganic carbon-free rings containing alkaline cations

Michelle Hernández-Arganis, Simón Hernández-Ortega, Rubén A. Toscano, Verónica García-Montalvo and Raymundo Cea-Olivares*

The alkali metal–crown ether ‘misfit’ is used as a novel synthetic strategy for the preparation of discrete inorganic rings containing the unusual rubidium– and caesium–soft chalcogenide bonds.

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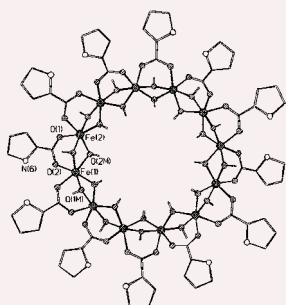


An inorganic iron complex that inhibits wild-type and an isoniazid-resistant mutant 2-*trans*-enoyl-ACP (CoA) reductase from *Mycobacterium tuberculosis*

Jaim S. Oliveira, Eduardo H. S. Sousa, Luiz A. Basso,* Moisés Palaci, Reynaldo Dietze, Diógenes S. Santos and Ícaro S. Moreira*

The inactivation of both wild-type and I21V InhA enzymes by $[\text{Fe}^{\text{II}}(\text{CN})_5(\text{INH})]^{3-}$ requires neither activation by KatG nor the presence of NADH, and its mechanism of action probably involves interaction with the NADH binding pocket of the enoyl reductases.

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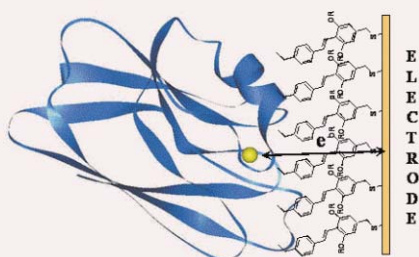


An Fe(III) wheel with a zwitterionic ligand: the structure and magnetic properties of $[\text{Fe}(\text{OMe})_2(\text{proline})]_{12}[\text{ClO}_4]_{12}$

Abd-Alhakeem H. Abu-Nawwas, Joan Cano, Paul Christian, Talal Mallah, Gopalan Rajaraman, Simon J. Teat, Richard E. P. Winpenny* and Yasuhiko Yukawa*

A planar dodecanuclear iron ring is reported which forms cation:anion stacks with alternate layers of perchlorate counter-ions.

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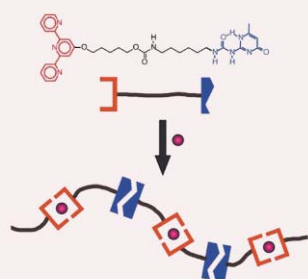


Fast, long-range electron-transfer reactions of a ‘blue’ copper protein coupled non-covalently to an electrode through a stilbenyl thiolate monolayer

Fraser A. Armstrong, Nicola L. Barlow, Paul L. Burn, Kevin R. Hoke, Lars J. C. Jeuken, Catherine Shenton and Graham R. Webster

A stilbenyl thiol forms a self-assembled monolayer on a gold electrode that extends the range of fast electron transfer with adsorbed protein molecules to over 15 Å.

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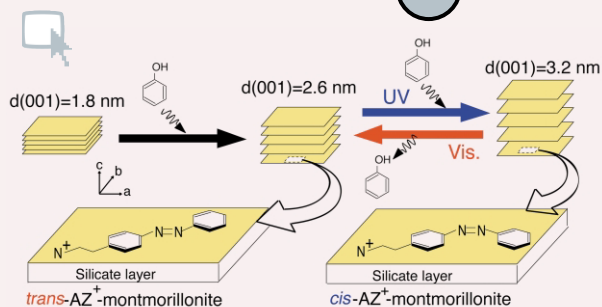


New supramolecular polymers containing both terpyridine metal complexes and quadruple hydrogen bonding units

Harald Hofmeier, Abdelkrim El-ghayoury, Albertus P. H. J. Schenning and Ulrich S. Schubert*

For the first time, two different strong non-covalent interactions were combined in one molecular moiety by designing a molecule containing both a terpyridine and an ureidopyrimidinone unit. Hydrogen-bonded coordination polymers containing metal complexes were obtained, as indicated by UV-vis and $^1\text{H-NMR}$ spectroscopy as well as viscosimetry.

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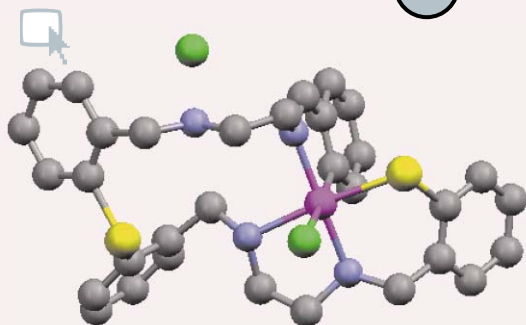


Photocontrol of the adsorption behavior of phenol for an azobenzene-montmorillonite intercalation compound

Tomohiko Okada,* Yusuke Watanabe and Makoto Ogawa*

Phenol was intercalated into *p*-(ω -dimethylhydroxyethyl-ammonioethoxy)azobenzene (AZ⁺)-montmorillonite intercalation compound by UV irradiation and was deintercalated by subsequent visible light irradiation.

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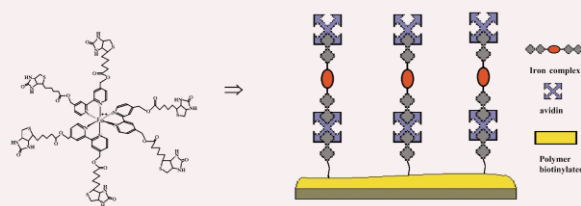


Contrasting coordination behaviour of 22-membered chalcogenaza (Se, Te) macrocycles towards Pd(II) and Pt(II): Isolation and structural characterization of the first metallamacrocyle with a C–Pt–Se linkage

Snigdha Panda, Harkesh B. Singh* and Ray J. Butcher

The synthesis and crystal structure of a novel 23-membered Pt(IV) metallamacrocyclic complex with a C–Pt–Se linkage is reported.

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A new biotinylated tris bipyridinyl iron(II) complex as redox biotin-bridge for the construction of supramolecular biosensing architectures

Naoufel Haddour, Chantal Gondran and Serge Cosnier*

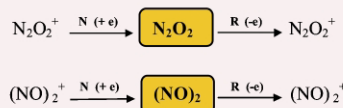
A new tris(bipyridyl)iron(II) complex bearing six 3 D oriented biotin groups has been designed and applied to the anchoring of multilayered avidin structures on an electrode surface.

326

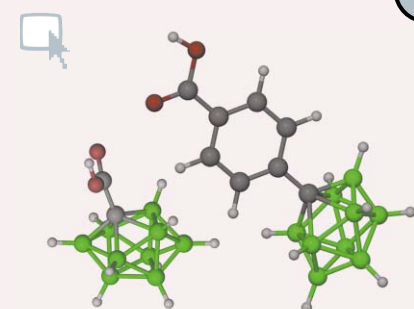
Discovery of two high-energy N₂O₂ isomers

Giulia de Petris,* Fulvio Cacace* and Anna Troiani

Two N₂O₂ isomers containing N₂/O₂ and NO/NO subunits, respectively, were detected by neutralization-reionization mass spectrometry (NRMS) as metastable species with lifetimes exceeding 1 μ s.



328

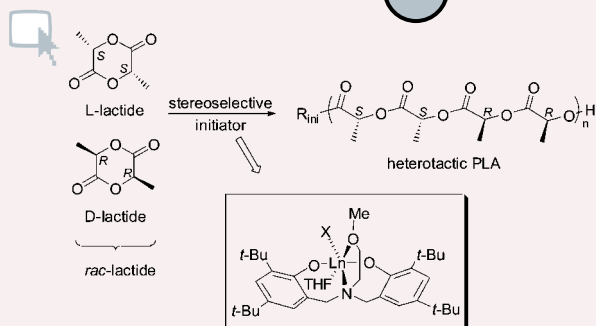


Polyhedral monocarbaborane chemistry. Carboxylic acid derivatives of the [closo-2-CB₉H₁₀]⁻ anion

Andreas Franken, Colin A. Kilner and John D. Kennedy*

The new [closo-2-CB₉H₉-2-(COOH)]⁻ and [closo-2-CB₉H₉-2-(C₆H₄-*para*-COOH)]⁻ anions, obtained in overall 45–50% yield from B₁₀H₁₄, are potentially useful synthons for the further derivatisation of {closo-CB₉} monocarbaboranes.

330

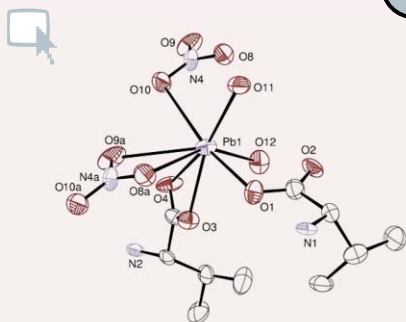


Stereoselective ring-opening polymerization of racemic lactide using alkoxy-amino-bis(phenolate) group 3 metal complexes

Chen-Xin Cai, Abderramane Amgoune, Christian W. Lehmann and Jean-François Carpentier*

Alkyl- and amido-yttrium complexes of a non-chiral tetradentate alkoxy-amino-bis(phenolate) ligand initiate the fast and controlled polymerization of *rac*-lactide to give heterotactic-rich polylactic acid.

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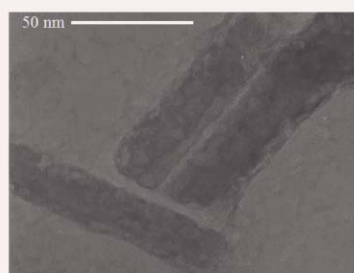


Definitive identification of lead(II)-amino acid adducts and the solid state structure of a lead–valine complex

Neil Burford,* Melanie D. Eelman, Wesley G. LeBlanc, T. Stanley Cameron and Katherine N. Robertson

Electrospray ionization mass spectra of lead(II) nitrate–amino acid mixtures provide unequivocal identification of lead species with each of the essential amino acids and assignment of a valine complex has been supported with the first crystallographic characterization of a lead–amino acid complex.

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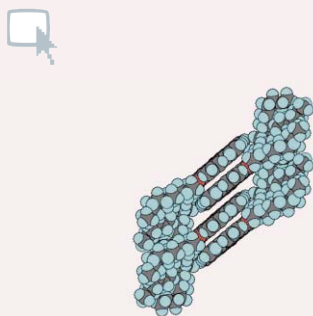


Indium sulfide nanorods from single-source precursor

Mohammad Afzaal, Mohammad A. Malik and Paul O'Brien*

Thin films comprised of In_2S_3 nanorods have been prepared on glass substrates by metal-organic chemical vapour deposition [$\text{Et}_2\text{In}(\text{S}_2\text{CNMe}^t\text{Bu})$] without either template or catalyst.

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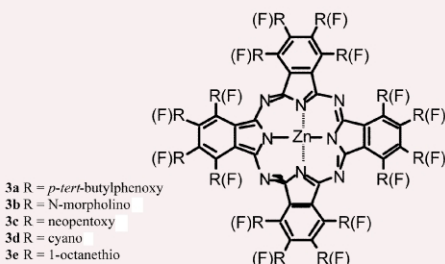


Partially stripped insulated nanowires: a lightly substituted hexa-*peri*-hexabenzocoronene-based columnar liquid crystal

Zhaohui Wang, Mark D. Watson, Jishan Wu and Klaus Müllen*

Despite carrying only three alkyl chains at a single peripheral focal area, a new hexa-*peri*-hexabenzocoronene derivative forms stable columnar liquid crystalline mesophases and is easily processable from solution or from the melt, the latter due to a practically accessible isotropization temperature.

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Polysubstituted phthalocyanines by nucleophilic substitution reactions on hexadecafluorophthalocyanines

Clifford C. Leznoff* and José L. Sosa-Sanchez

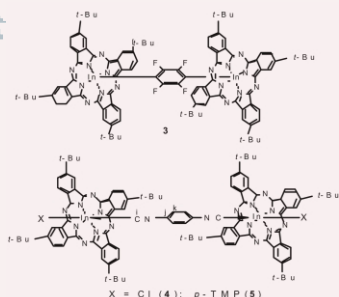
Nucleophilic substitution reactions on hexadecafluorophthalocyaninato zinc(II) with a variety of O, N, C, and S nucleophiles led to narrowly defined mixtures of polysubstituted phthalocyanines.

340

Excited state properties of monomeric and dimeric axially bridged indium phthalocyanines upon UV-Vis laser irradiation

Yu Chen, Danilo Dini, Michael Hanack,* Mamoru Fujitsuka and Osamu Ito

Defined photoexcited states in the dimeric indium phthalocyanines **3** - **5** can be formed *via* irradiation at both off-resonance as well as resonant frequencies in the UV-Visible range. Linear and nonlinear optical properties of **3** - **5** were determined and the optical limiting effect was analyzed in terms of structural modifications.

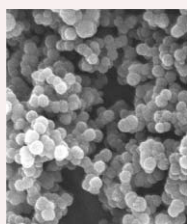


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A chemical route from PTFE to amorphous carbon nanospheres in supercritical water

Xiaogang Yang, Cun Li, Wei Wang, Baojun Yang, Shuyuan Zhang and Yitai Qian*

Poly(tetrafluoroethylene) can be decomposed into amorphous carbon nanospheres *via* supercritical water treatment in an autoclave at 550 °C, which may be applied to deal with other halogenous polymer wastes.

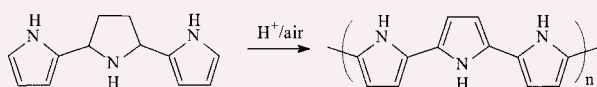


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Formation of polypyrrole from 2,5-bis(2-pyrrolyl)pyrrolidine

Arnd Garsuch, Rita R. Sattler and Peter G. Pickup*

2,5-Bis(2-pyrrolyl)pyrrolidine has been found to spontaneously polymerize under acidic conditions in the presence of air to produce fully aromatic polypyrrole.

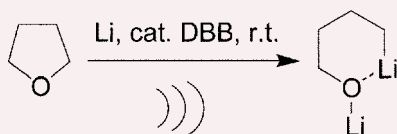


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Unexpected cleavage of tetrahydrofuran by catalytic reductive lithiation

Stéphane Streiff, Nigel Ribeiro and Laurent Désaubry*

DBB, an electron transporter, can open THF at room temperature under sonication without any Lewis acid activation. This feature was successfully exploited in the straightforward synthesis of bis-silanes.

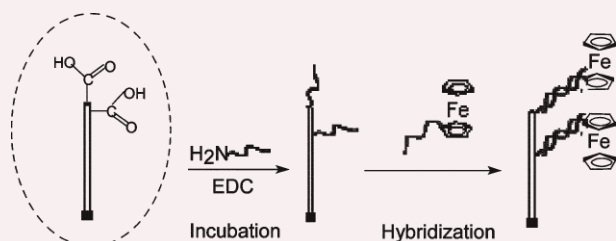


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Aligned carbon nanotube-DNA electrochemical sensors

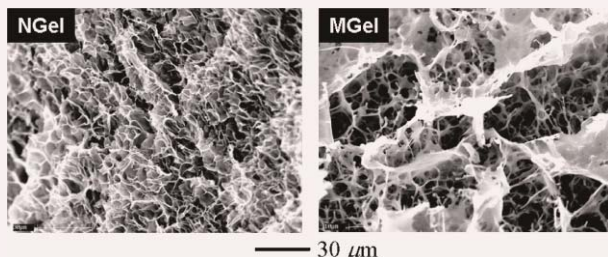
Pingang He and Liming Dai*

Single-strand DNA chains were chemically grafted onto aligned carbon nanotube electrodes, leading to novel aligned carbon nanotube-DNA sensors of a high sensitivity and selectivity for probing complementary DNA and target DNA chains of specific sequences.



Preparation of thermosensitive PNIPAAm hydrogels with superfast response

Xian-Zheng Zhang and Chih-Chang Chu*



A simple effective strategy was developed to prepare thermosensitive poly(*N*-isopropylacrylamide) (PNIPAAm) hydrogel with a superfast response by using THF as a foaming agent as well as a precipitation agent during the polymerization/crosslinking in water at room temperature. The superfast response PNIPAAm hydrogel may contribute significantly to the construction of novel intelligent devices in the future.

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