

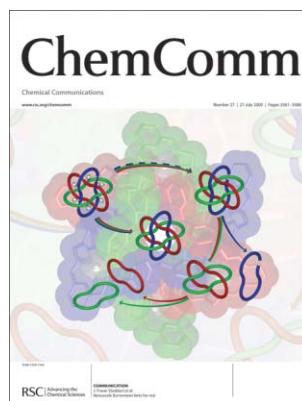
IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (27) 3361–3488 (2005)



Cover

See Graham J. Hutchings *et al.*, page 3385. The Au/Fe₂O₃ catalyst achieves target conversion and selectivity for the competitive oxidation of dilute CO in the presence of moist excess H₂ and CO₂. Image reproduced by permission of Philip Landon, Jonathan Ferguson, Benjamin E. Solsona, Tomas Garcia, Albert F. Carley, Andrew A. Herzing, Christopher J. Kiely, Stanislaw E. Golunski and Graham J. Hutchings, *Chem. Commun.*, 2005, 3385.



Inside cover

See J. Fraser Stoddart *et al.*, page 3394. Fully demetallated molecular Borromean links are realised. Image reproduced by permission of Andrea J. Peters, Kelly S. Chichak, Stuart J. Cantrill and J. Fraser Stoddart, *Chem. Commun.*, 2005, 3394.

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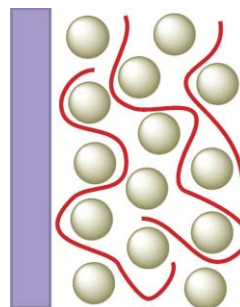
FEATURE ARTICLE

3375

Metal nanoparticle—conjugated polymer nanocomposites

Bryan C. Sih and Michael O. Wolf*

Nanocomposites in which metal nanoparticles are embedded in conjugated polymers are intriguing materials with interesting electronic and optical properties. This review highlights recent developments in the preparation and properties of these hybrid materials.



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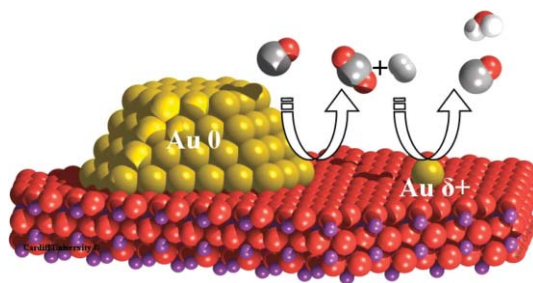
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Selective oxidation of CO in the presence of H₂, H₂O and CO₂ via gold for use in fuel cells

Philip Landon, Jonathan Ferguson, Benjamin E. Solsona, Tomas Garcia, Albert F. Carley, Andrew A. Herzing, Christopher J. Kiely, Stanislaw E. Golunski and Graham J. Hutchings*

An Au/Fe₂O₃ catalyst prepared using a two-stage calcination procedure achieves target conversion and selectivity for the competitive oxidation of dilute CO in the presence of moist excess H₂ and CO₂.

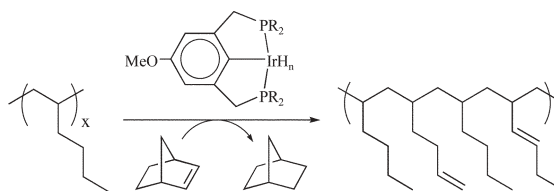


3388

Dehydrogenation of aliphatic polyolefins catalyzed by pincer-ligated iridium complexes

Amlan Ray, Keming Zhu, Yury V. Kissin, Anna E. Cherian, Geoffrey W. Coates* and Alan S. Goldman*

We report the first example of the catalytic dehydrogenation of aliphatic polyolefins to give partially unsaturated hydrocarbon polymers.

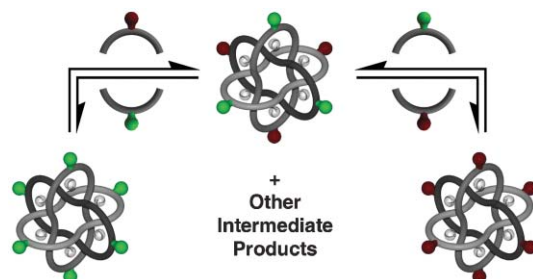


3391

Dynamic nanoscale Borromean links

Kelly S. Chichak, Stuart J. Cantrill and J. Fraser Stoddart*

Employing halogen atom labels on one of the ligand precursors, the lability of at least some of the 30 dative and 12 imine bonds stabilizing and constituting the three rings of a metallo-Borromean linked compound are scrambled in acidic methanolic solution.

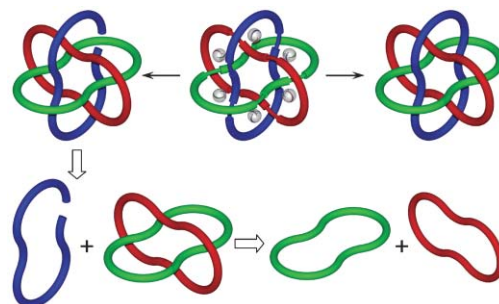


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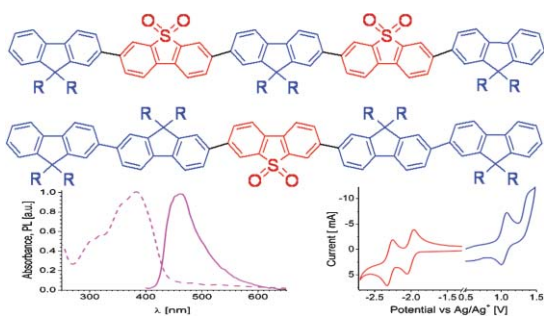
Nanoscale Borromean links for real

Andrea J. Peters, Kelly S. Chichak, Stuart J. Cantrill and J. Fraser Stoddart

In the midst of reducing and demetallating a Borromean Ring (BR) complex to give a neutral BR compound, a chemical proof of the BR topology was obtained.



3397

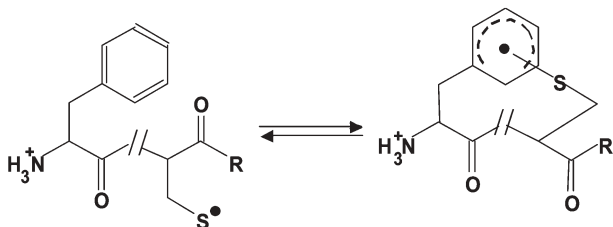


Dibenzothiophene-*S,S*-dioxide–fluorene co-oligomers. Stable, highly-efficient blue emitters with improved electron affinity

Irene I. Perepichka, Igor F. Perepichka,* Martin R. Bryce* and Lars-Olof Pålsson

New hybrid dibenzothiophene-*S,S*-dioxide oligomers have been synthesised and shown to be highly-efficient blue fluorophores which can be reversibly p- and n-doped.

3400

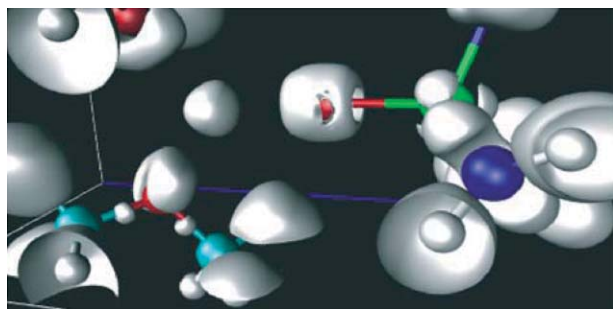


Intramolecular addition of cysteine thiol radicals to phenylalanine in peptides: formation of cyclohexadienyl type radicals

Thomas Nauser, Giulio Casi, Willem H. Koppenol and Christian Schöneich*

The addition of a cysteine thiol radical to phenylalanine yields a substituted cyclohexadienyl radical. In the presence of oxygen or other oxidants, this reaction may represent a novel pathway to thioether cross-linked peptides and proteins.

3403

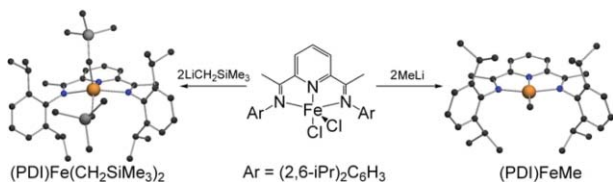


Synthesis of a four-coordinate titanium(IV) oxoanion via deprotonation and decarbonylation of complexed formate

Arjun Mendiratta, Joshua S. Figueroa and Christopher C. Cummins*

An oxoanion of titanium(IV) has been synthesized by deprotonation of bound formate followed by spontaneous decarbonylation. Quantum chemical calculations illuminate the nature of the Ti–O titanoxo bond.

3406

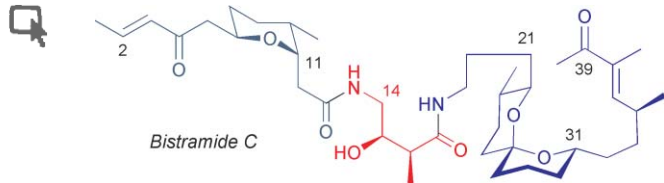


Square planar bis(imino)pyridine iron halide and alkyl complexes

Marco W. Bouwkamp, Suzanne C. Bart, Eric J. Hawrelak, Ryan J. Trovitch, Emil Lobkovsky and Paul J. Chirik*

Synthesis and characterization of square planar iron chloride and methyl complexes with bis(imino)pyridine ligands are described; treatment of the corresponding ferrous dihalide with two equivalents of $\text{LiCH}_2\text{SiMe}_3$ afforded the bis(alkyl) complex.

3421

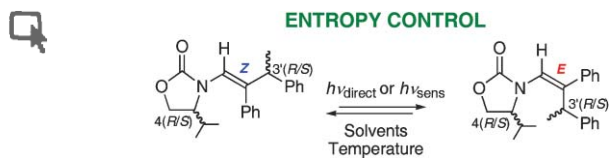


Total synthesis and structure validation of (+)-bistramide C

Peter Wipf* and Tamara D. Hopkins

The first total synthesis of the marine natural product (+)-bistramide C confirms the *a priori* assignments of its relative and absolute configurations, which were originally derived from the combined use of $[\alpha]_D$ analysis, NMR, and synthesis.

3424

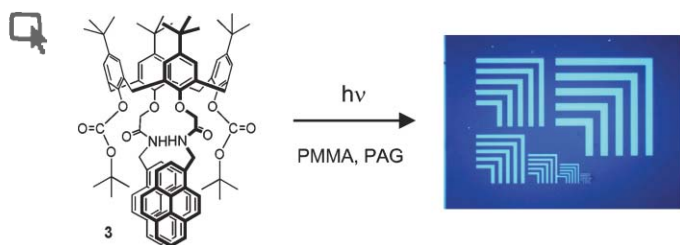


Stereoselective *E/Z* photoisomerization of oxazolidinone functionalized enecarbamates: direct and triplet sensitized irradiation

Hideaki Saito, J. Sivaguru, Steffen Jockusch, Yoshihisa Inoue,* Waldemar Adam* and Nicholas J. Turro*

Oxazolidinone-functionalized enecarbamates undergo diastereoselective *E/Z* photoisomerization upon direct and triplet sensitized irradiations with chiral/achiral sensitizers, showing the enhanced product diastereoselectivity dependent on the solvent and temperature.

3427

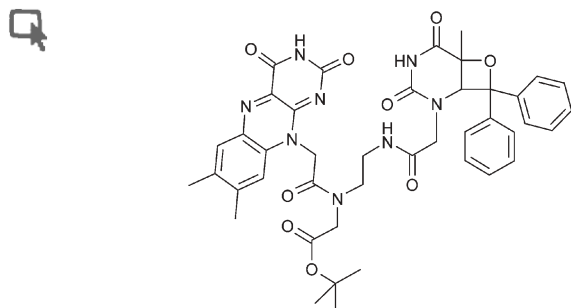


An excimer emission approach for patterned fluorescent imaging

Jong-Man Kim,* Sung Jun Min, Soon W. Lee, Ju Han Bok and Jong Seung Kim*

A selective removal of *t*-Boc protecting groups of a pyrene-containing calixarene derivative **3** in a polymer film by chemical amplification technique allowed generation of patterned fluorescence images in the film without employing wet developing processes.

3430



(6-4)-Photolyase activity requires a charge shift reaction

Thorsten Stafforst and Ulf Diederichsen*

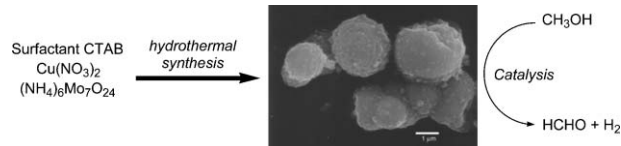
A model compound containing a thymine oxetane moiety linked to a flavin chromophore was investigated regarding (6-4)-photolyase activity. The need for a charge shift reaction was demonstrated by a detailed pH-dependent kinetic analysis.

3433

Surfactant-promoted novel reductive synthesis of supported metallic Cu nanoclusters and their catalytic performances for selective dehydrogenation of methanol

Rajaram Bal, Mizuki Tada and Yasuhiro Iwasawa*

We have found a surfactant-promoted novel reductive synthesis of metallic Cu nanoclusters supported on metal oxides under hydrothermal synthesis conditions, which are active for the selective dehydrogenation of methanol to formaldehyde and hydrogen.

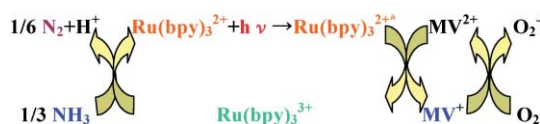


3436

Visible light decomposition of ammonia to dinitrogen by a new visible light photocatalytic system composed of sensitizer (Ru(bpy)₃²⁺), electron mediator (methylviologen) and electron acceptor (dioxxygen)

Masao Kaneko,* Naoto Katakura, Chihiro Harada, Yoshihito Takei and Mikio Hoshino

Visible light decomposition of aqueous NH₃ to N₂ was achieved by using a new photocatalytic system based on a molecular photoelectron relay.

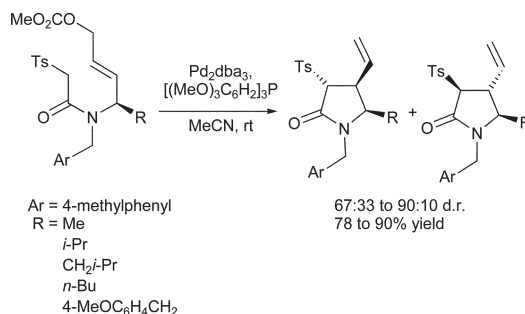


3439

Stereoselective γ -lactam synthesis via palladium-catalysed intramolecular allylation

Donald Craig,* Christopher J. T. Hyland and Simon E. Ward

Amino acid-derived allylic carbonates possessing α -tosylacetamide moieties undergo ambient-temperature, stereoselective Pd(0)-catalysed cyclisation in good yield and with high 3,4-*syn* stereoselectivity.

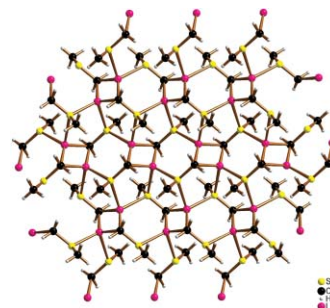


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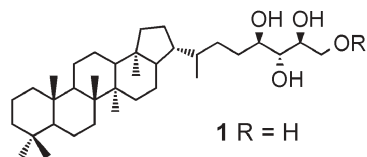
Solvent-free methylthiomethylithium [LiCH₂SMe]_∞: solid state structure and thermal decomposition

Kai Ruth, Robert E. Dinnebier,* Stefan W. Tönnies, Edith Alig, Ingeborg Sanger, Hans-Wolfram Lerner and Matthias Wagner*

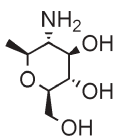
The solid state structure of solvent-free [LiCH₂SMe]_∞ was determined by high-resolution X-ray powder diffraction. The compound violently explodes upon heating to $T = 160 \pm 5^\circ\text{C}$ under an argon atmosphere.



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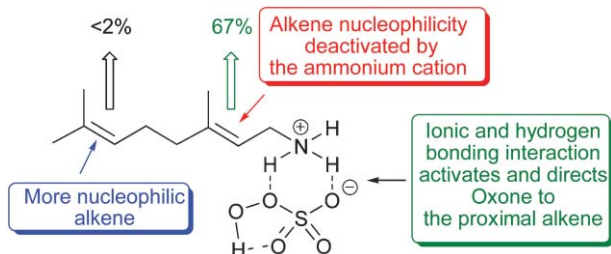


Concise syntheses of bacteriohopanetetrol and its glucosamine derivative

Weidong Pan, Yongmin Zhang, Guangyi Liang, Stéphane P. Vincent* and Pierre Sinaÿ*

Short syntheses of bacteriohopanetetrol **1** and its glucosamine derivative **2** are described. The efficiency of these syntheses relies on the coupling of a ribitol derivative to the hopane skeleton *via* its activation through the adequate organocopper derivative.

3448

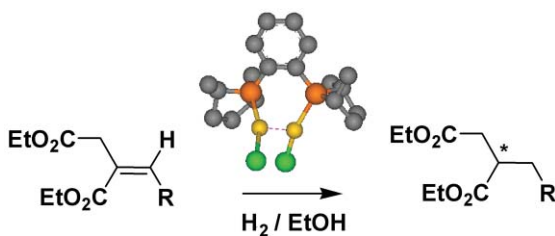


Highly regioselective and diastereoselective epoxidation of allylic amines with Oxone

Varinder K. Aggarwal* and Guang Yu Fang

Allylic amines (as their protonated ammonium salts) can be epoxidised with high *syn* diastereoselectivity and regioselectivity at the proximal alkene in substrates with several double bonds using Oxone.

3451

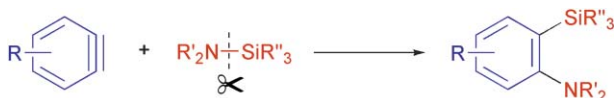


Enantioselective hydrogenation of alkenes and imines by a gold catalyst

Camino González-Arellano, Avelino Corma,* Marta Iglesias and Felix Sánchez

A new neutral dimeric gold(I) complex bearing the [(*R,R*)-Me-Duphos] ligand has been synthesized. The gold complex catalyzes the asymmetric hydrogenation of alkenes and imines under mild reaction conditions.

3454



Aminosilylation of arynes with aminosilanes: synthesis of 2-silylaniline derivatives

Hiroto Yoshida,* Takashi Minabe, Joji Ohshita and Atsutaka Kunai*

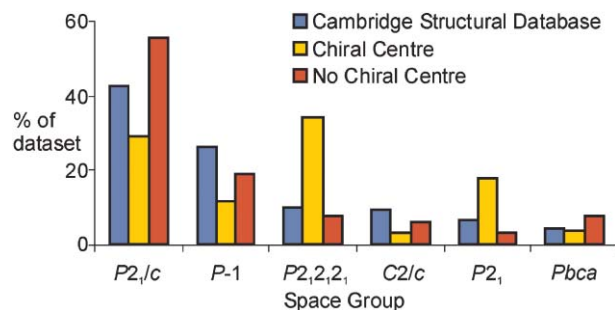
A variety of 2-silylaniline derivatives were synthesized straightforwardly *via* a novel aminosilylation reaction, where the nitrogen-silicon σ -bond of aminosilanes added across the carbon-carbon triple bond of arynes.

3457

Achiral molecules in non-centrosymmetric space groups

Elna Pidcock*

A database survey of organic molecules indicates molecular flexibility plays a role in determining whether an achiral molecule crystallises displaying a centrosymmetric or non-centrosymmetric space group.

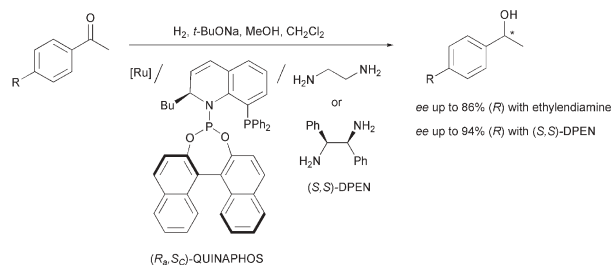


3460

Ruthenium-catalysed asymmetric hydrogenation of ketones using QUINAPHOS as the ligand

Simon Burk, Giancarlo Franciò and Walter Leitner*

Highly enantioselective ruthenium-catalysed hydrogenation of aromatic ketones is achieved with (R_a, S_C) -QUINAPHOS in the presence of achiral and chiral diamines as co-catalysts.

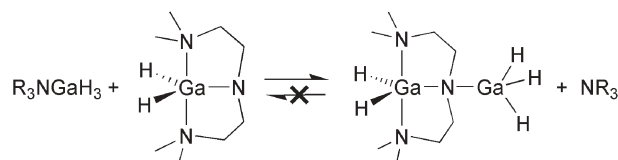


3463

Mono- and digallane complexes of a tridentate amido-diamine ligand

Bing Luo, Benjamin E. Kucera and Wayne L. Gladfelter*

The synthesis and characterization of $\text{H}_2\text{GaN}(\text{CH}_2\text{CH}_2\text{NMe}_2)_2$ (**1**) and $\text{H}_2\text{GaN}(\text{CH}_2\text{CH}_2\text{NMe}_2)_2\text{GaH}_3$ (**2**) are reported. **1** is a potentially useful precursor to gallium-containing films and **2** exhibits an unusual structural feature.

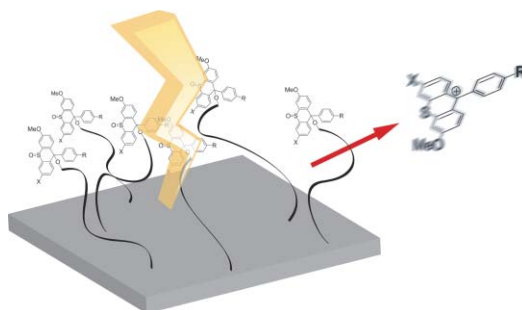


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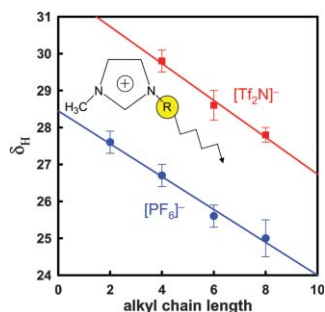
S(O)-Pixyl protecting group as efficient mass-tag

Pablo L. Bernad, Jr, Safraz Khan, Vladimir A. Korshun, Edwin M. Southern and Mikhail S. Shchepinov*

S(O)-pixyls and novelty trityl systems with adjustable stability: used as mass-tags and protecting groups.



3469

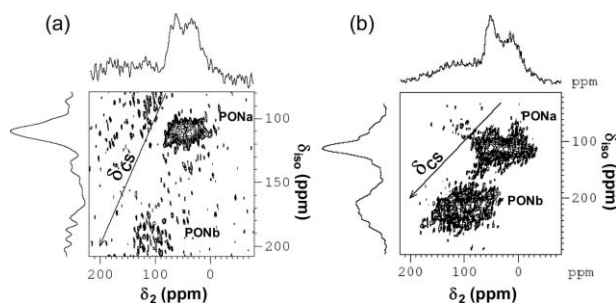


The Hildebrand solubility parameters, cohesive energy densities and internal energies of 1-alkyl-3-methylimidazolium-based room temperature ionic liquids

Sang Hyun Lee and Sun Bok Lee*

The Hildebrand solubility parameters, cohesive energy densities and internal energies of 1-alkyl-3-methylimidazolium-based room temperature ionic liquids were determined by the intrinsic viscosity method and their dependencies on the length of the alkyl group analyzed.

3472

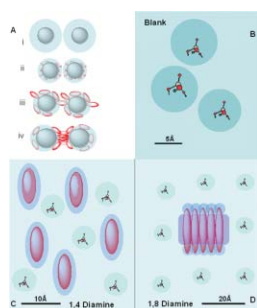


A very sensitive high-resolution NMR method for quadrupolar nuclei: SPAM-DQF-STMAS

Jean-Paul Amoureux,* Alexandrine Flambard, Laurent Delevoye and Lionel Montagne

The SPAM-STMAS method allows observing in 17 hours (b) ^{17}O spectra of $30\text{Nb}_2\text{O}_5-70\text{NaPO}_3$ that are hardly observable in 48 hours (a) with SPAM-3QMAS.

3475

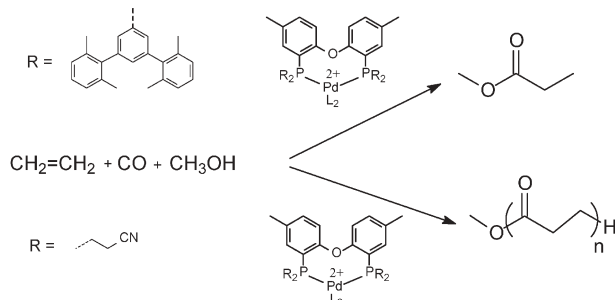


Putrescine homologues control silica morphogenesis by electrostatic interactions and the hydrophobic effect

David Belton, Siddharth V. Patwardhan and Carole C. Perry*

A systematic model study on the role(s) of putrescine homologues on silicification is presented and it is proposed that electrostatic forces between additive and silicic acid, and the hydrophobic behaviour of the additives are both important in silicification.

3478



New diphosphine ligands based on diphenyl ether for the Pd-catalyzed CO/ethene copolymerization

Maria Caporali, Christian Müller, Bastiaan B. P. Staal, Duncan M. Tooke, Anthony L. Spek and Piet W. N. M. van Leeuwen*

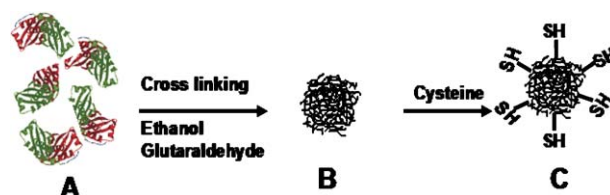
The catalytic activity and selectivity of palladium(II) complexes of new, flexible bidentate ligands in the CO/ethene copolymerization reaction have been found to change considerably with the steric properties of the ligands.

3481

Enzyme nanoparticles-based electronic biosensor

Guodong Liu, Yuehe Lin,* Veronika Ostatná and Joseph Wang*

A simple and effective method to prepare an enzyme electronic biosensor by immobilizing enzyme nanoparticles directly onto the gold electrode surface is described. Immobilized enzyme nanoparticles retain their redox and electrocatalytic activities and were used to develop reagentless biosensors for H_2O_2 detection without promoters and mediators.

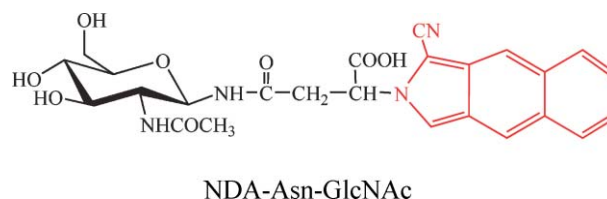


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Resolution of *N*-linked oligosaccharides in glycoproteins based upon transglycosylation reaction by CE-TOF-MS

Jun Zhe Min, Toshimasa Toyo'oka,* Masaru Kato and Takeshi Fukushima

The resolution of asparagine-type oligosaccharides in glycoproteins was carried out by combination of the transglycosylation reaction using Endo-M and CE-TOF-MS.



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