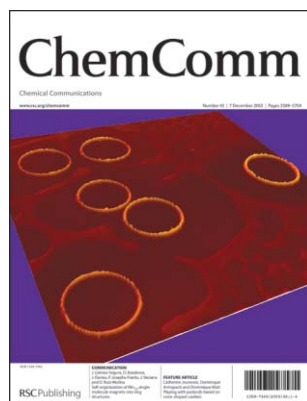


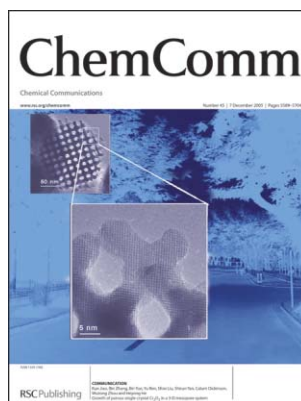
IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (45) 5589-5704 (2005)



Cover

See Jordi Gómez-Segura, Olga Kazakova, Julia Davies, Patrick Josephs-Franks, Jaume Veciana and Daniel Ruiz-Molina, page 5615. Cooling evaporation of a CH_2Cl_2 solution of Mn_{12} clusters on HOPG has been used to induce their self-organization into magnetic rings. Image reproduced with the permission of the Controller of HMSO and the Queen's Printer for Scotland, from *Chem. Commun.*, 2005, 5615.



Inside cover

See Kun Jiao, Bin Zhang, Bin Yue, Yu Ren, Shixi Liu, Shirun Yan, Calum Dickinson, Wuzong Zhou and Heyong He, page 5618. Porous single crystals of Cr_2O_3 were fabricated using mesoporous KIT-6 as a hard template. Image reproduced by permission of Wuzong Zhou *et al.*, from *Chem. Commun.*, 2005, 5618.

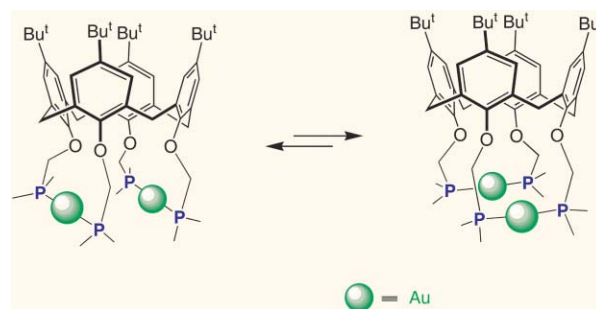
FEATURE ARTICLE

5603

Playing with podands based on cone-shaped cavities. How can a cavity influence the properties of an appended metal centre?

Catherine Jeunesse, Dominique Armspach and Dominique Matt*

The potential of molecules that combine the properties of a conical cavity with those of a covalently-linked transition-metal centre is highlighted through the assessment of cyclodextrin- and calixarene-derived podands ("cavitand" ligands) in coordination chemistry and catalysis.



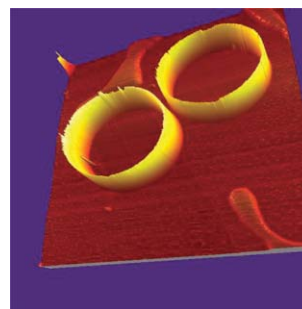
COMMUNICATIONS

5615

Self-organization of Mn_{12} single-molecule magnets into ring structures induced by breath-figures as templates

Jordi Gómez-Segura, Olga Kazakova, Julia Davies, Patrick Josephs-Franks, Jaume Veciana and Daniel Ruiz-Molina*

Cooling evaporation of a CH_2Cl_2 solution of Mn_{12} clusters on HOPG initiates the formation of water droplets that act as templates for the formation of self-assembled molecular magnetic rings.



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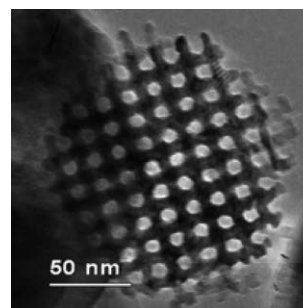
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5618

Growth of porous single-crystal Cr₂O₃ in a 3-D mesopore system

Kun Jiao, Bin Zhang, Bin Yue, Yu Ren, Shixi Liu, Shirun Yan, Calum Dickinson, Wuzong Zhou* and Heyong He*

Single-crystal Cr₂O₃ with regular mesopores has been synthesized using mesoporous silica KIT-6 as a template and characterized by using XRD, HRTEM and nitrogen adsorption/desorption.

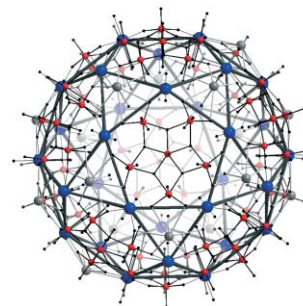


5621

Ferrimagnetically ordered nanosized polyoxomolybdate-based cluster spheres

Bogdan Botar,* Paul Kögerler,* Achim Müller, Ricardo Garcia-Serres and Craig L. Hill*

Step-wise substitution of two different magnetic heterometal cations in a spherical keplerate capsule is achieved under reducing conditions. Ferrimagnetic interactions are observed for the first time for this cluster type.

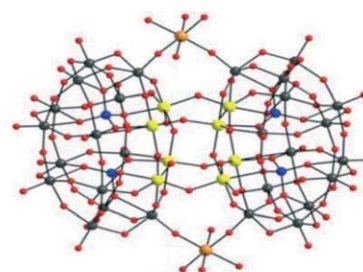


5624

Coordination chemistry of the hexavacant tungstophosphate [H₂P₂W₁₂O₄₈]¹²⁻: synthesis and characterization of iron(III) complexes derived from the unprecedented {P₂W₁₄O₅₄} fragment

Béatrice Godin, Jacqueline Vaissermann, Patrick Herson, Laurent Ruhlmann, Michel Verdaguer and Pierre Gouzerh*

The cluster [H₁₂P₄W₂₈Fe₈O₁₂₀]¹⁶⁻ derives from the previously unobserved tetravacant Dawson anion [P₂W₁₄O₅₄]¹⁴⁻ and displays the rare cubic Fe₈ topology. It acts as a ligand for two Co(II) ions in [{Co(H₂O)₄]₂{H₁₂P₄W₂₈Fe₈O₁₂₀}]¹²⁻.

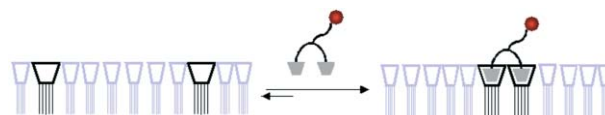


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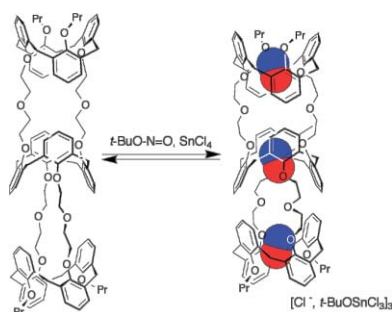
Dynamic multivalent recognition of cyclodextrin vesicles

Choon Woo Lim, Bart Jan Ravoo* and David N. Reinhoudt*

Cyclodextrin vesicles have dynamic host membranes that recognize guest molecules through multivalent host-guest interaction reminiscent of multivalent binding of a ligand with receptors in a biological membrane.



5630

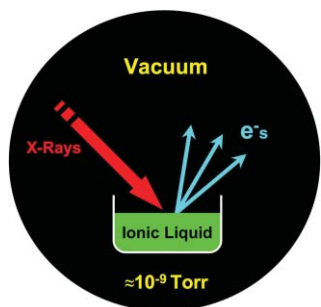


A procedure for filling calixarene nanotubes

Valentina Sgarlata, Voltaire G. Organo and Dmitry M. Rudkevich*

Alkyl nitrites readily transfer nitrosonium into calixarene-based synthetic nanotubes thus offering a mild, effective and precise method to fill them.

5633

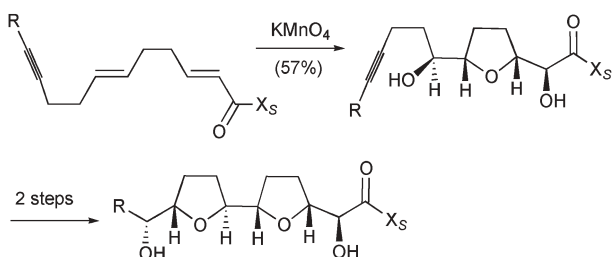


Ionic liquids *in vacuo*; solution-phase X-ray photoelectron spectroscopy

Emily F. Smith, Ignacio J. Villar Garcia, David Briggs and Peter Licence*

The *in situ* monitoring of catalysis in room temperature ionic liquid (RTIL) solutions is fundamental to the understanding of catalytic processes and the role of RTILs in catalytic turnover; we describe how XPS can be used to give information on both pure RTILs and catalytically-active RTIL-based solutions.

5636

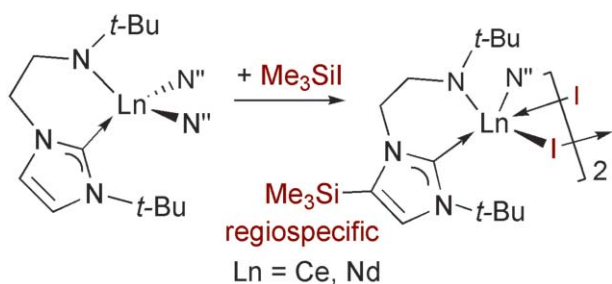


A metal-oxo mediated approach to the synthesis of 21,22-diepi-membrarollin

Yulai Hu and Richard C. D. Brown*

The asymmetric synthesis of 21,22-diepi-membrarollin (**14**) is reported. Key features include the use of highly selective oxidative cyclisations mediated by metal-oxo species to form the six stereogenic centres present in the *bis*-THF core.

5638



Regioselective C–H activation of lanthanide-bound N-heterocyclic carbenes

Polly L. Arnold* and Stephen T. Liddle

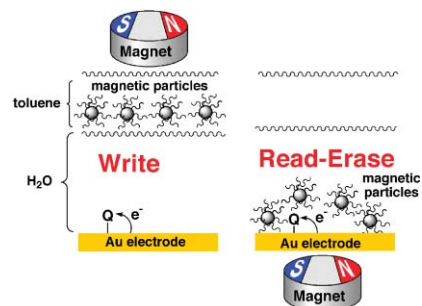
The reaction of $\text{Ln}(\text{L})\text{N}''_2$ ($\text{Ln} = \text{Nd, Ce}$; $\text{L} = t\text{-BuNCH}_2\text{CH}_2[\text{C}\{\text{NCHCHN}t\text{-Bu}\}]$, $\text{N}'' = \text{N}(\text{SiMe}_3)_2$) with trimethylsilyl iodide regioselectively functionalises the carbene backbone at the C4-carbene ring position to afford the silylated complex $\text{Ln}(\text{L}')\text{N}''\text{I}$; $\text{Ln}(\text{L}')\text{N}''_2$ is also reported ($\text{L}' = t\text{-BuNCH}_2\text{CH}_2[\text{C}\{\text{NC}(\text{SiMe}_3)\text{CHN}t\text{-Bu}\}]$).

5641

A quinone-functionalized electrode in conjunction with hydrophobic magnetic nanoparticles acts as a “Write–Read–Erase” information storage system

Eugenii Katz and Itamar Willner*

Integration of hydrophobic magnetic nanoparticles with a quinone-functionalized Au electrode in a water–toluene two-phase assembly yields a “Write–Read–Erase” information processing system.

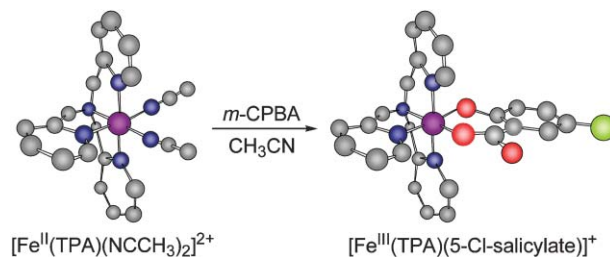


5644

Self-hydroxylation of perbenzoic acids at a nonheme iron(II) center

Na Young Oh, Mi Sook Seo, Mi Hee Lim, Mark B. Consugar, Mi Joo Park, Jan-Uwe Rohde, Jaehong Han, Kwan Mook Kim, Jinheung Kim, Lawrence Que, Jr* and Wonwoo Nam*

Treatment of mononuclear nonheme iron(II) complexes bearing two *cis*-labile sites with perbenzoic acids results in the self-hydroxylation of the aromatic ring to form the corresponding iron(III)–salicylate complexes.

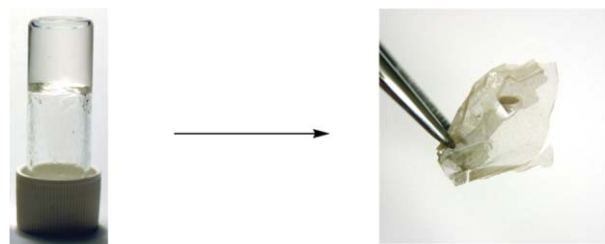


5647

Robust gels created using a self-assembly and covalent capture strategy

Christine S. Love, Victor Chechik, David K. Smith,* Ian Ashworth and Colin Brennan

The self-assembly of dendritic building blocks containing multiple terminal alkenes on their surfaces yields soft gel-phase materials – subsequent Grubbs’ metathesis leads to covalent cross-linking between the alkenes and the formation of robust swellable gels.

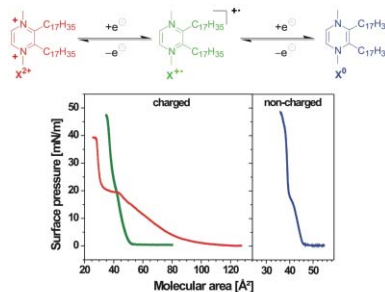


5650

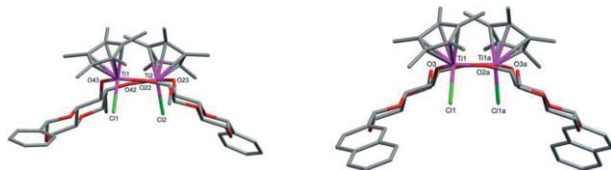
N,N'-Dimethyl-2,3-dialkylpyrazinium salts as redox-switchable surfactants? Redox, spectral, EPR and surfactant properties

Michael Schmittl,* Mukul Lal, Karlheinz Graf,* Gunnar Jeschke, Irina Suske and Josef Salbeck

Pyrazinium salts ($X^0 \rightleftharpoons X^{2+}$) may become useful novel redox-switchable surfactant systems that can even be complemented by the third redox stage (X^{2+}), if some restrictions are accommodated.



5653

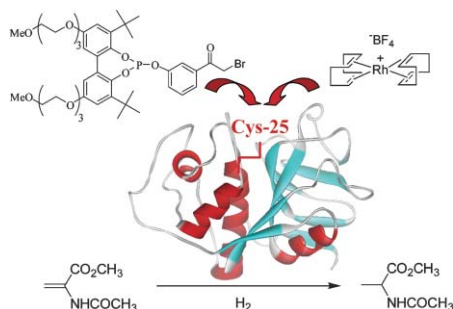


Sugar ligands in organotitanium complexes

Daniela Küntzer, Lars Jessen and Jürgen Heck*

Using ligands derived from D-glucose, dinuclear organotitanium compounds with interesting structural features were synthesized.

5656

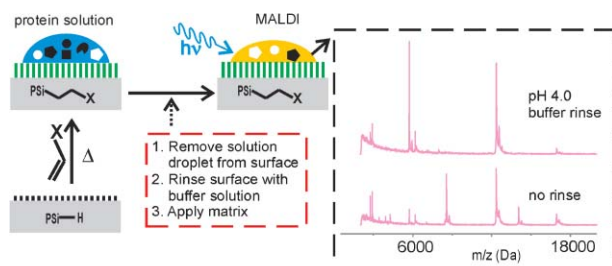


Merging homogeneous catalysis with biocatalysis; papain as hydrogenation catalyst

Lavinia Panella, Jaap Broos, Jianfeng Jin, Marco W. Fraaije, Dick B. Janssen, Margot Jeronimus-Stratingh, Ben L. Feringa, Adriaan J. Minnaard and Johannes G. de Vries*

Papain, modified at Cys-25 with a monodentate phosphite ligand and complexed with Rh(COD)₂BF₄, is an active catalyst in the hydrogenation of methyl 2-acetamidoacrylate.

5659

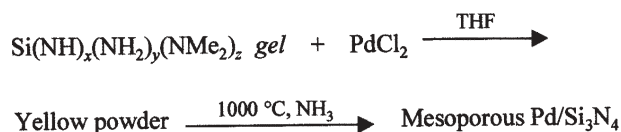


Functionalized porous silicon surfaces as MALDI-MS substrates for protein identification studies

Tadesse Z. Mengistu, Leroi DeSouza and Sylvie Morin*

Alkyl monolayer modified porous silicon functional surfaces are employed for selective binding of proteins from complex mixtures (through washing of the deposited mixture spot using appropriate buffer) and MALDI-MS is used to detect the components retained on the surface.

5662



General method of preparation of mesoporous M/Si₃N₄ nano-composites via a non-aqueous sol-gel route

Fei Cheng,* Stephen M. Kelly, Nigel A. Young, Stephen Clark, M. Grazia Francesconi, Frédéric Lefebvre and John S. Bradley

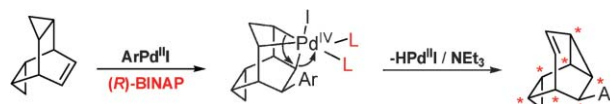
A high-surface-area mesoporous Pd/Si₃N₄ nanocomposite is obtained by pyrolysis of a silicon palladium imide-chloride complex which was synthesized by reaction of silicon diimide gel with palladium chloride.

5665

Palladium-catalyzed, stereoselective rearrangement of a tetracyclic allyl cyclopropane under arylation

Jörg Storsberg, Min-Liang Yao, Nüket Öcal, Armin de Meijere, Arnold E. W. Adam and Dieter E. Kaufmann*

The first example of a stereoselective π,σ domino-Heck reaction under concomitant rearrangement of the tetracyclic allylcyclopropane *endo,exo*-bishomobarrelene proceeds via an intramolecular insertion reaction into a cyclopropane σ -bond, generating 7 chiral centers.

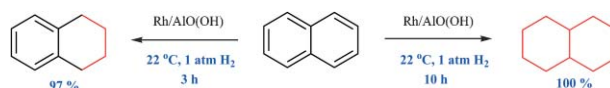


5667

Rhodium nanoparticles entrapped in boehmite nanofibers: recyclable catalyst for arene hydrogenation under mild conditions

In Soo Park, Min Serk Kwon, Namdu Kim, Jae Sung Lee, Kyung Yeon Kang and Jaiwook Park*

A new recyclable rhodium catalyst was synthesized by a simple procedure from readily available reagents, which showed high activities in the hydrogenation of various arenes under 1 atm H_2 at room temperature.

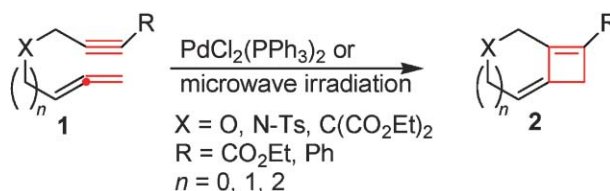


5670

Highly efficient [2 + 2] intramolecular cyclizations of allenynes under microwave irradiation: construction of fused bicyclic compounds

Chang Ho Oh,* Arun Kumar Gupta, Dai In Park and Nakjoong Kim

A Pd-catalyzed- and a microwave-mediated [2 + 2] cycloaddition of various 1,*n*-allenynes were developed. Particularly, the microwave-irradiated [2 + 2] cycloaddition of allenynes can provide a simple, general, and eco-friendly synthetic method to fused bicyclo[*m*,2,0]alkadienes.

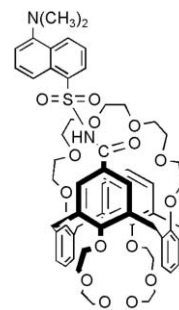


5673

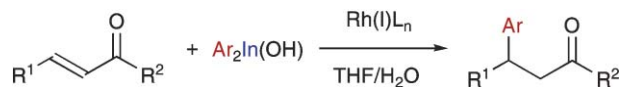
Novel fluorogenic calix[4]arene-bis(crown-6-ether) for selective recognition of thallium(I)

Galina G. Talanova,* Ebony D. Roper, Nicole M. Buie, Maryna G. Gorbunova, Richard A. Bartsch and Vladimir S. Talanov

A new fluorogenic, dansyl group-containing derivative of 1,3-*alternate* calix[4]arene-bis(crown-6-ether) embodies the first example of a calixarene-based fluorescent chemosensor for selective recognition of Tl(I).



5676

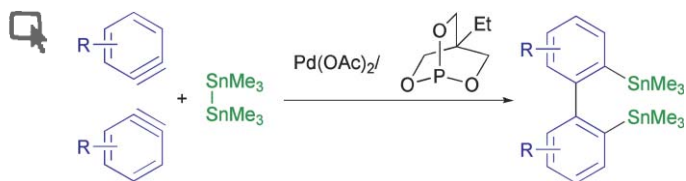


Rhodium-catalysed 1,4-addition of diaryliindium hydroxides to α,β -unsaturated carbonyl compounds

Tomoya Miura and Masahiro Murakami*

Diaryliindium(III) hydroxides react with α,β -unsaturated carbonyl compounds in the presence of a rhodium catalyst to afford the 1,4-addition products, demonstrating the utility of diaryliindium(III) hydroxide as an aryl source with rhodium catalysts.

5678

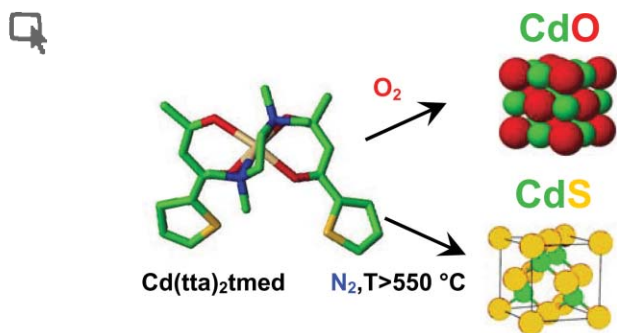


Palladium-catalysed dimerisation–distannylation of arynes: synthesis and reaction of 2,2'-distannylbiaryls

Hiroto Yoshida,* Kenji Tanino, Joji Ohshita and Atsutaka Kunai*

Biaryl skeletons were assembled from arynes and distannanes depending upon the palladium-bicyclic phosphite complex-catalysed dimerisation–distannylation reaction. The resulting 2,2'-distannylbiaryls were convertible into variously substituted biaryls by the use of their carbon–tin bonds.

5681

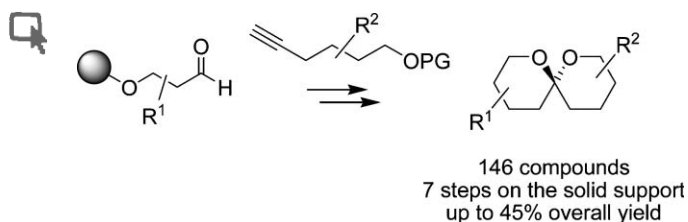


Multifunctional cadmium single source precursor for the selective deposition of CdO or CdS by a solution route

Graziella Malandrino,* Sebastiana T. Finocchiaro, Patrizia Rossi, Paolo Dapporto and Ignazio L. Fragalà

The novel adduct $\text{Cd}(\text{tta})_2 \cdot \text{tmed}$ represents a multipurpose single source, and has been reproducibly and selectively applied to the preparation of nanostructured CdS or CdO.

5684



Solid phase synthesis of a spiro[5.5]ketal library

Stefan Sommer and Herbert Waldmann

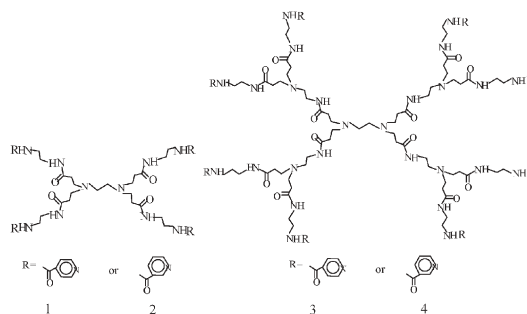
A spiro[5.5]ketal library embodying the core structure of numerous biologically active natural products was synthesized employing a double intramolecular hetero Michael reaction as a key transformation.

5687

A 7.72% efficient dye sensitized solar cell based on novel necklace-like polymer gel electrolyte containing latent chemically cross-linked gel electrolyte precursors

Li Wang, Shibi Fang,* Yuan Lin, Xiaowen Zhou and Miyu Li

Novel necklace-like polymer gel electrolytes containing latent chemically cross-linked gel electrolyte precursors were prepared for quasi-solid dye sensitized solar cells with a highest efficiency of 7.72% and an active area of 0.25 cm² under AM1.5 condition at 100 mW cm⁻² irradiation.

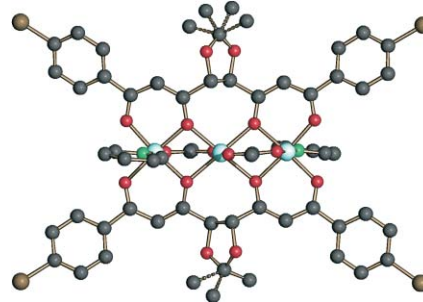


5690

Structural diversity in the assembly of helicate-type nickel(II) complexes with enantiopure bis(β -diketonate) ligands

Markus Albrecht,* Sabrina Dehn, Gerhard Raabe and Roland Fröhlich

Structural diversity is observed in the formation of dinuclear and trinuclear nickel(II) complexes of chiral bis(β -diketonate) ligands and is investigated by different analytical methods.

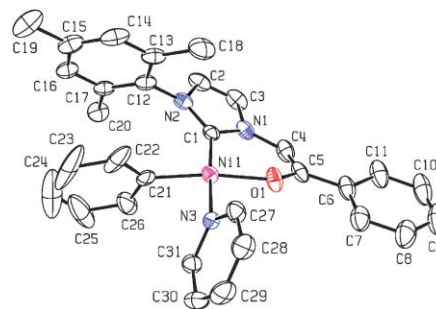


5693

Synthesis, structure, and olefin polymerization with nickel(II) N-heterocyclic carbene enolates

Benjamin E. Ketz, Xavier G. Ottenwaelder and Robert M. Waymouth*

Two novel N-heterocyclic carbene enolate nickel complexes have been prepared and shown to be active for ethylene and propylene polymerization to yield linear polymers.

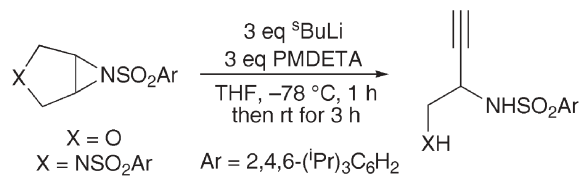


5696

Organolithium-mediated conversion of β -functionalised aziridines into alkynyl amino alcohols and diamines

Jianhui Huang and Peter O'Brien*

Dihydrofuran and dihydropyrrole *N*-triisopropylbenzenesulfonyl aziridines are transformed into alkynyl amino alcohols and diamines respectively using 3 equiv. *sec*-butyllithium–PMDETA in THF; use of *n*-butyllithium–(–)-sparteine in Et₂O gave an alkynyl amino alcohol in 60% ee.



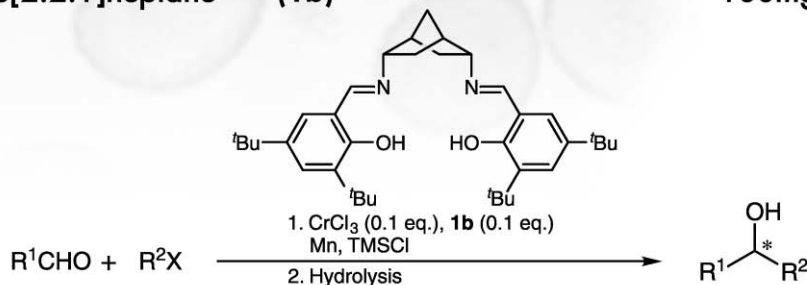
Hidekazu Miyaji, Gilles Gasser, Stephen J. Green, Yann Molard, Sharon M. Strawbridge and James H. R. Tucker

Selective electrochemical sensing of acidic organic molecules *via* a novel guest-to-host proton transfer reaction

DIANANE- Salen Ligand -for Asymmetric Nozaki-Hiyama-Kishi Reaction-

(1*R*,2*R*,4*R*,5*R*)-2,5-Bis(3,5-di-*tert*-butyl-2-hydroxybenzylideneamino)-
bicyclo[2.2.1]heptane (1a) 100mg [B2652]

(1*S*,2*S*,4*S*,5*S*)-2,5-Bis(3,5-di-*tert*-butyl-2-hydroxybenzylideneamino)-
bicyclo[2.2.1]heptane (1b) 100mg [B2653]



DIANANE-Salen ligand **1** is a useful reagent for the asymmetric Nozaki-Hiyama-Kishi (NHK) Reaction as a ligand for transition metal complexes. Such complexes can be used as highly enantioselective catalysts to obtain chiral secondary alcohols in high enantiopurity.

A. Berkessel, D. Menche, C. A. Sklorz, M. Schröder, I. Paterson, *Angew. Chem. Int. Ed.*, **42**, 1032 (2003);
I. Paterson, H. Bergmann, D. Menche, A. Berkessel, *Org. Lett.*, **6**, 1293 (2004).

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AUTHOR INDEX

- Adam, Arnold E. W., 5665
Albrecht, Markus, 5690
Armspach, Dominique, 5603
Arnold, Polly L., 5638
Ashworth, Ian, 5647
Bartsch, Richard A., 5673
Botar, Bogdan, 5621
Bradley, John S., 5662
Brennan, Colin, 5647
Briggs, David, 5633
Broos, Jaap, 5656
Brown, Richard C. D., 5636
Buie, Nicole M., 5673
Chechik, Victor, 5647
Cheng, Fei, 5662
Clark, Stephen, 5662
Consugar, Mark B., 5644
Dapporto, Paolo, 5681
Davies, Julia, 5615
Dehn, Sabrina, 5690
de Meijere, Armin, 5665
DeSouza, Leroi, 5659
de Vries, Johannes G., 5656
Dickinson, Calum, 5618
Fang, Shibi, 5687
Feringa, Ben L., 5656
Finocchiaro, Sebastiana T., 5681
Fraaije, Marco W., 5656
Fragalà, Ignazio L., 5681
Francesconi, M. Grazia, 5662
Fröhlich, Roland, 5690
Garcia-Serres, Ricardo, 5621
Godin, Béatrice, 5624
Gómez-Segura, Jordi, 5615
Gorbunova, Maryna G., 5673
Gouzerh, Pierre, 5624
Graf, Karlheinz, 5650
Gupta, Arun Kumar, 5670
Han, Jaehong, 5644
He, Heyong, 5618
Heck, Jürgen, 5653
Herson, Patrick, 5624
Hill, Craig L., 5621
Hu, Yulai, 5636
Huang, Jianhui, 5696
Janssen, Dick B., 5656
Jeronimus-Stratingh, Margot, 5656
Jeschke, Gunnar, 5650
Jessen, Lars, 5653
Jeunesse, Catherine, 5603
Jiao, Kun, 5618
Jin, Jianfeng, 5656
Josephs-Franks, Patrick, 5615
Kang, Kyung Yeon, 5667
Katz, Eugenii, 5641
Kaufmann, Dieter E., 5665
Kazakova, Olga, 5615
Kelly, Stephen M., 5662
Ketz, Benjamin E., 5693
Kim, Jinheung, 5644
Kim, Kwan Mook, 5644
Kim, Nakjoong, 5670
Kim, Namdu, 5667
Kögerler, Paul, 5621
Kunai, Atsutaka, 5678
Küntzer, Daniela, 5653
Kwon, Min Serk, 5667
Lal, Mukul, 5650
Lee, Jae Sung, 5667
Lefebvre, Frédéric, 5662
Li, Miyu, 5687
Licence, Peter, 5633
Liddle, Stephen T., 5638
Lim, Choon Woo, 5627
Lim, Mi Hee, 5644
Lin, Yuan, 5687
Liu, Shixi, 5618
Love, Christine S., 5647
Malandrino, Graziella, 5681
Matt, Dominique, 5603
Mengistu, Tadesse Z., 5659
Minnaard, Adriaan J., 5656
Miura, Tomoya, 5676
Morin, Sylvie, 5659
Müller, Achim, 5621
Murakami, Masahiro, 5676
Nam, Wonwoo, 5644
O'Brien, Peter, 5696
Öcal, Nüket, 5665
Oh, Chang Ho, 5670
Oh, Na Young, 5644
Ohshita, Joji, 5678
Organo, Voltaire G., 5630
Ottawaelder, Xavier G., 5693
Panella, Lavinia, 5656
Park, Dai In, 5670
Park, In Soo, 5667
Park, Jaiwook, 5667
Park, Mi Joo, 5644
Que, Jr, Lawrence, 5644
Raabe, Gerhard, 5690
Ravoo, Bart Jan, 5627
Reinhoudt, David N., 5627
Ren, Yu, 5618
Rohde, Jan-Uwe, 5644
Roper, Ebony D., 5673
Rossi, Patrizia, 5681
Rudkevich, Dmitry M., 5630
Ruhlmann, Laurent, 5624
Ruiz-Molina, Daniel, 5615
Salbeck, Josef, 5650
Schmittel, Michael, 5650
Seo, Mi Sook, 5644
Sgarlata, Valentina, 5630
Smith, David K., 5647
Smith, Emily F., 5633
Sommer, Stefan, 5684
Storsberg, Jörg, 5665
Suske, Irina, 5650
Talanov, Vladimir S., 5673
Talanova, Galina G., 5673
Tanino, Kenji, 5678
Vaisermann, Jacqueline, 5624
Veciana, Jaume, 5615
Verdaguer, Michel, 5624
Villar Garcia, Ignacio J., 5633
Waldmann, Herbert, 5684
Wang, Li, 5687
Waymouth, Robert M., 5693
Willner, Itamar, 5641
Yan, Shirun, 5618
Yao, Min-Liang, 5665
Yoshida, Hiroto, 5678
Young, Nigel A., 5662
Yue, Bin, 5618
Zhang, Bin, 5618
Zhou, Wuzong, 5618
Zhou, Xiaowen, 5687

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