#### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (2) 141-288 (2005)



#### Cover

Self assembly of catechol imine ligands. See p. 157.



#### Inside cover

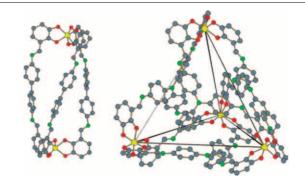
Circular hexagonal, concentric lamellar and other unique structures possible with silica wires. See p. 166.

#### FEATURE ARTICLE

#### Catechol imine ligands: from helicates to supramolecular tetrahedra

Markus Albrecht,\* Ingo Janser and Roland Fröhlich

Catechol imines represent versatile ligands with special conformational features which allow the self-assembly of metallosupramolecular coordination compounds like helicates or 4:4 tetrahedra.



#### COMMUNICATIONS

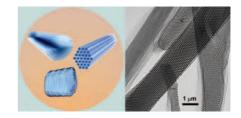
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#### Hierachical mesoporous silica wires by confined assembly

Donghai Wang, Rong Kou, Zhenglong Yang, Jibao He, Zhenzhong Yang\* and Yunfeng Lu\*

The assembly of silicate and surfactant confined within cylindrical alumina pore channels results in circular hexagonal, concentric lamellar and other unique mesostructures.



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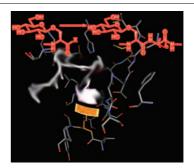
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### "Polar patch" proteases as glycopeptiligases

Katie J. Doores and Benjamin G. Davis\*

Screening of proteases with 'polar patch' chemical modifications has revealed new biocatalysts capable of native glycopeptide enzymatic ligation.

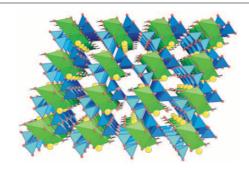


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#### A novel microporous copper silicate: Na<sub>2</sub>Cu<sub>2</sub>Si<sub>4</sub>O<sub>11</sub>·2H<sub>2</sub>O

Paula Brandão, Filipe A. Almeida Paz and João Rocha\*

Synthesis and structural characterisation of a thermally stable novel three-dimensional microporous copper silicate open-framework.

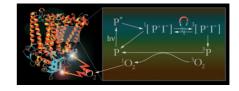


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#### Magnetic field effect on singlet oxygen production in a biochemical system

Yan Liu, Ruth Edge, Kevin Henbest, Christiane R. Timmel, P. J. Hore\* and Peter Gast

The yield of singlet oxygen sensitized by chemically modified, carotenoidless bacterial photosynthetic reaction centres and the ensuing oxidative damage are both shown to be magnetic field-dependent.

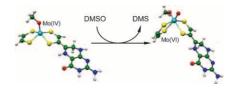


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#### Promotion of oxygen atom transfer in Mo and W enzymes by bicyclic forms of the pterin cofactor

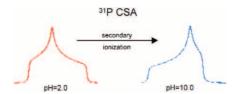
Jonathan P. McNamara, John A. Joule, Ian H. Hillier\* and C. David Garner

Density functional theory calculations show that protonated forms of bicyclic structures of the "molybdopterin" can facilitate oxygen atom transfer at the Mo (or W) centre of oxotransferase enzymes.



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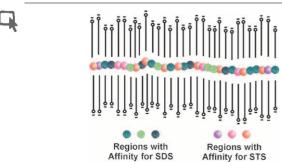


Straightforward detection of the secondary ionisation of the phosphate group and pK determinations by high-resolution solid-state  $^{31}P$  NMR

Carole Gardiennet, Bernard Henry, Paul Kuad, Bernard Spiess and Piotr Tekely\*

The suitability of high-resolution solid-state  $^{31}P$  NMR for a straightforward determination of the protonation state of phosphate groups as well as of their p $K_2$  values extracted from solid state mono: dianionic ratios has been demonstrated.

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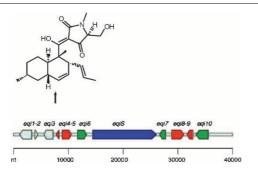
Separation of denatured proteins in free solution on a microchip based on differential binding of alkyl sulfates with different carbon chain lengths

Chang Lu,\* Aaron E. Smith and Harold G. Craighead\*

Denatured proteins were separated in free solution on a microfluidic chip without any sieving matrix after carrying out the denaturation with a mixture of alkyl sulfates with different carbon chain lengths.

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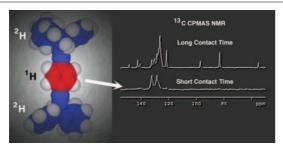


Equisetin biosynthesis in Fusarium heterosporum

James W. Sims, John P. Fillmore, Douglas D. Warner and Eric W. Schmidt\*

A tetramic acid biosynthetic gene cluster was characterized and includes EqiS, a hybrid PKS-NRPS from a filamentous fungus.

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Highlighting gyroscopic motion in crystals in <sup>13</sup>C CPMAS spectra by specific isotopic substitution and restricted cross polarization

Steven D. Karlen and Miguel A. Garcia-Garibay\*

Carbon atoms undergoing dynamic exchange in crystals of molecular gyroscopes can be uncovered from strongly overlapping signals with <sup>13</sup>C CPMAS NMR by specific isotopic substitution and selective cross polarization.

¬ P

#### Pre-organization induced synthesis of a crossed alkenebridged nisin Z DE-ring mimic by ring-closing metathesis

Nourdin Ghalit, Dirk T. S. Rijkers, Johan Kemmink, Cees Versluis and Rob M. J. Liskamp\*

The preferred formation of the bicyclic crossed alkene-bridged mimic of the DE-bisthioether-ring system of the antibiotic nisin hints at a favorable pre-organization of the linear RCM-precursor peptide.

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### A peptide-catalyzed asymmetric Stetter reaction

Steven M. Mennen, Jarred T. Blank, Michelle B. Tran-Dubé, Jason E. Imbriglio and Scott J. Miller\*

Thiazolylalanine, in appropriately functionalized form, has been found to function as an enantioselective catalyst for an intramolecular Stetter reaction (up to 81% ee).

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## Highly selective epoxidation of styrene using a transition metal-aluminium(III) complex containing the [MeAl(2-py)<sub>3</sub>] anion (2-py = 2-pyridyl)

Carmen Soria Alvarez, Felipe García, Simon M. Humphrey, Alexander D. Hopkins,\* Richard A. Kowenicki, Mary McPartlin, Richard A. Layfield, Robert Raja,\* Michael C. Rogers, Anthony D. Woods and Dominic S. Wright\*

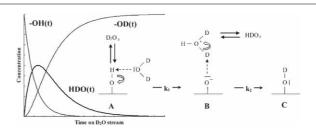
The reactions of [MeAl(2-py)<sub>3</sub>Li·thf] (1) with FeCl<sub>2</sub> or  $Cp_2Mn$  in toluene give the heterometallic complexes [{MeAl(2-py)<sub>3</sub>}<sub>2</sub>M] [M = Fe (2), Mn (3)]; complex 2 has been shown to be a highly selective styrene epoxidation catalyst in air. 201

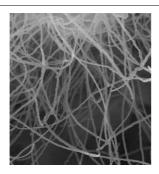
## Direct quantitative determination of surface Brønsted acidity of solids by H/D exchange using D<sub>2</sub>O

Nicolas Keller, Guillaume Koehl, François Garin and Valérie Keller\*

A highly flexible method for direct and quantitative determination of surface Brønsted acidity of solids in terms of number, type and strength of the acid sites based on quantitative H/D exchange kinetics between the acid solid and gaseous  $D_2O$  has been developed and applied to materials covering the whole range of acidity.







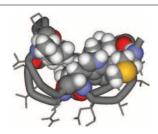
#### Doped spiral alumina nanowires

Nien-Fang Wu, Hung-Jen Chen, Yu-Lun Chueh, Su-Jien Lin, Li-Jen Chou and Wen-Kuang Hsu\*

Doped spiral alumina nanowires are produced *via* annealing of high entropy alloys. Nanowires emit blue–green light, together with a small PL peak centered at yellow wavelength.

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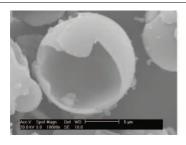


### A small peptide stereochemically customized as a globular fold with a molecular cleft

Soumendra Rana, Bijoy Kundu and Susheel Durani\*

A boat shaped peptide molecular fold is generated by stereochemical modification of a 20-residue  $\beta$ -hairpin peptide, making it a promising prototype for future optimization as a molecular receptor.

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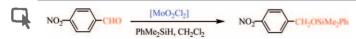


## Synthesis of siliceous hollow spheres with large mesopore wall structure by supercritical CO<sub>2</sub>-in-water interface templating

Jiawei Wang, Yongde Xia, Wenxin Wang, Robert Mokaya\* and Martyn Poliakoff

Mesostructured hollow silica spheres (see images) may be synthesised *via* CO<sub>2</sub>-in-water emulsion templating in the presence of PEO-PPO-PEO block copolymers under supercritical fluid conditions.

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### [MoO<sub>2</sub>Cl<sub>2</sub>] as catalyst for hydrosilylation of aldehydes and ketones

Ana C. Fernandes, Ricardo Fernandes, Carlos C. Romão and Beatriz Royo\*

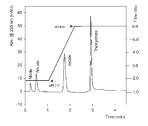
The high valent molybdenum-dioxo complex [MoO<sub>2</sub>Cl<sub>2</sub>] catalyzes the addition of dimethylphenylsilane to aromatic aldehydes and also ketones to afford the corresponding dimethylphenylsilyl ethers.

#### 215

### Double gradient ion chromatography on a short carboxybetaine coated monolithic anion exchanger

Brett Paull,\* Colmán Ó Ríordáin and Pavel N. Nesterenko

Work describes the technique of 'double gradient ion chromatography', based upon using flow gradients on short monolithic type anion exchangers, combined with eluent composition gradients, and simultaneously improving selectivity and reducing run times.

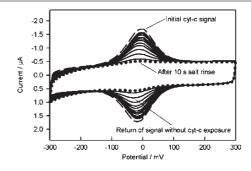


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## Unexpected retention of electrostatically adsorbed cytochrome c in high ionic strength solutions

Cory M. DiCarlo and David L. Compton\*

Redox inactivation, but not removal, of electrostatically adsorbed cytochrome c (cyt-c) on alkanethiol modified gold surfaces was observed after exposure to salt solution. Careful consideration must, therefore, be used to avoid mistaking deactivation for desorption of the protein from the electrode.



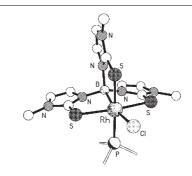
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### The first rhodaboratrane: $[RhCl(PPh_3)\{B(mt)_3\}](Rh \rightarrow B)$ (mt = methimazolyl)

Ian R. Crossley, Mark R. St.-J. Foreman, Anthony F. Hill,\* Andrew J. P. White and David J. Williams

The reaction of  $[Rh(C_6H_5)Cl_2(PPh_3)_2]$  with  $Na[HB(mt)_3]$  (mt = methimazolyl) provides  $[RhCl(PPh_3)\{B(mt)_3\}](Rh \rightarrow B)$  the first authentic example of a compound with a rhodium-boron dative bond.



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## A heme-like, water-soluble iron(II) porphyrin: thermal and photoinduced properties, evidence for sitting-atop structure

Róbert Huszánk and Ottó Horváth\*

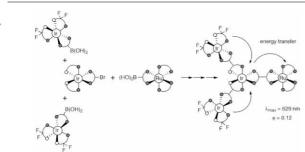
The thermal and photoinduced properties of the heme-like, water-soluble Fe(II)-tetrakis(4-sulfonatophenyl)porphyrin are characteristic of kinetically labile, sitting-atop complexes which exhibit irreversible porphyrin ligand to metal charge transfer.

### Novel amine-catalysed hydroalkoxylation reactions of activated alkenes and alkynes

Julie E. Murtagh, Séamus H. McCooey and Stephen J. Connon\*

Nucleophilic amines catalyse the 1,4-addition of alcohols to  $\alpha,\beta$ -unsaturated alkenes in good to excellent yields. The discovery of a nucleophile-catalysed dihydroalkoxylation reaction of an activated alkyne is also reported.

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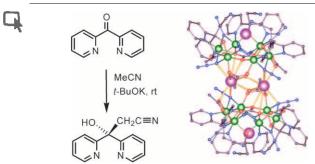


#### Boronic acid-substituted metal complexes: versatile building blocks for the synthesis of multimetallic assemblies

Kathryn J. Arm and J. A. Gareth Williams\*

Complexes based on  $[Ru(bpy)_3]^{2+}$  and  $[Ir(ppy)_2(bpy)]^+$  have been prepared, in which one bpy ligand incorporates a pendent boronic acid group. These compounds cross-couple with bromo-substituted complexes, offering a facile route to multimetallic assemblies.

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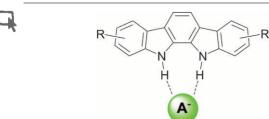


## A novel high-spin heterometallic $Ni_{12}K_4$ cluster incorporating large Ni-azide circles and an *in situ* cyanomethylated di-2-pyridyl ketone

Ming-Liang Tong,\* Montserrat Monfort, Juan Modesto Clemente Juan, Xiao-Ming Chen, Xian-He Bu, Masaaki Ohba and Susumu Kitagawa\*

The large ferromagnetically-coupled  $[Ni(\mu_{1,1}-N_3)]_6$  circles were trapped in a heterometallic  $Ni_{12}K_4$  cluster from the reaction of di-2-pyridyl ketone with nickel acetate and azide in the presence of potassium tert-butylate and an unprecedented  $in\ situ$  cyanomethylated ketone ligand was also generated.

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#### Indolocarbazoles: a new family of anion sensors

David Curiel, Andrew Cowley and Paul D. Beer\*

Simple preorganised indolo[2,3-a]carbazole derivatives are shown to recognise and sense anionic guest species using fluorescence spectroscopy.

A new class of long-wavelength fluorophores: strong red fluorescence, convenient synthesis and easy derivation

Yi Xiao, Fengyu Liu, Xuhong Qian\* and Jingnan Cui

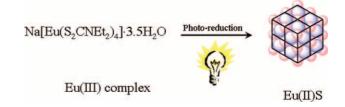
A new class of structurally simple fluorophores with strong long-wavelength emission have been developed through a very convenient synthetic procedure.

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A novel method for synthesizing EuS nanocrystals from a single-source precursor under white LED irradiation

Yasuchika Hasegawa, Mohammad Afzaal, Paul O'Brien,\* Yuji Wada and Shozo Yanagida\*

EuS nanocrystals, with an average diameter of 9 nm, have been synthesized by the photolysis of Na[Eu(S<sub>2</sub>CNEt<sub>2</sub>)<sub>4</sub>]·3.5H<sub>2</sub>O; the first quantum confined particles of EuS to be reported.

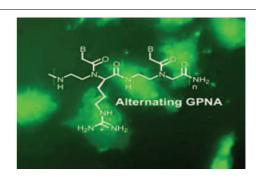


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Cell-permeable GPNA with appropriate backbone stereochemistry and spacing binds sequence-specifically to

Anca Dragulescu-Andrasi, Peng Zhou, Gaofei He and Danith H. Ly\*

Guanidine-based peptide nucleic acid (GPNA) with a D-backbone configuration and alternate spacing binds sequence-specifically to RNA and is readily taken up by both human somatic and embryonic stem (ES) cells.

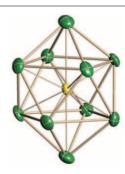


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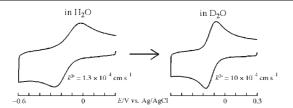
The closo-Pb<sub>10</sub><sup>2-</sup> Zintl ion in the [Ni@Pb<sub>10</sub>]<sup>2-</sup> cluster

Emren N. Esenturk, James Fettinger and Bryan Eichhorn\*

The cluster  $[Ni@Pb_{10}]^{2-}$ , prepared from  $Pb_9^{4-}$  and  $Ni(COD)_2$ , contains a new Zintl ion subunit, closo- $Pb_{10}^{2-}$ , centered by a Ni atom.



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### Notable deuterium effect on the electron transfer rate of myoglobin

Yasuhiro Mie,\* Chiho Yamada, Tadayuki Uno, Saburo Neya, Fumio Mizutani, Katsuhiko Nishiyama and Isao Taniguchi

A large 'inverse' kinetic isotope (deuterium) effect on the heterogeneous electron transfer reaction of wild-type myoglobin at an  $\rm In_2O_3$  electrode was observed with  $k_{\rm H2O}/k_{\rm D2O}=0.13$ .

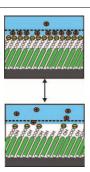
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### Anaerobic oxidation of non-activated secondary alcohols over $\text{Cu/Al}_2\text{O}_3$

Federica Zaccheria, Nicoletta Ravasio,\* Rinaldo Psaro and Achille Fusi

A copper catalyzed transfer dehydrogenation method allows quantitative transformation of aliphatic secondary alcohols into ketones under liquid phase, very mild experimental conditions.

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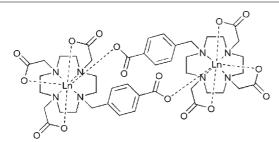
## Assembly modulates dissociation: electrokinetic experiments reveal peculiarities of the charge formation at monolayer films

Rüdiger Schweiss, Petra Welzel, Wolfgang Knoll and Carsten Werner\*

Self-assembled monomolecular films of alkanethiols chemisorbed on gold were used as a two-dimensional model system to study the effects of spatial confinement of surface functional groups on their acid-base behaviour.

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### pH Dependent self-assembly of dimetallic lanthanide complexes

Stephen Faulkner\* and Benjamin P. Burton-Pye

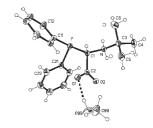
pH-Dependent self-association has been observed in a series of DO3A-derived lanthanide complexes bearing a carboxylate group that can act as a bridging ligand at high pH, switching on the luminescence from the lanthanide.



### Novel α-functionally substituted amino acids: diphenylphosphinoglycines

Joachim Heinicke,\* Normen Peulecke and Peter G. Jones

The three-component one-pot reaction of glyoxalic acid hydrate with Ph<sub>2</sub>PH and *t*BuNH<sub>2</sub> allows a convenient access to the first phosphinoglycines. The molecular structure, determined by X-ray crystallography, properties and some reactions are reported.



#### 265



### Evidence for a hydrogen abstraction mechanism in $P_{450}$ -catalyzed N-dealkylations

Mehul Bhakta, Paul F. Hollenberg and Kandatege Wimalasena\*

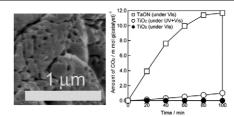
Series of N-cyclopropyl-N-(2-alkyl)cyclopropyl-p-chloroaniline and N-ethyl-N-fluoroethyl-p-chloroaniline derivatives were used to distinguish between the single electron transfer and the hydrogen atom transfer mechanism in the  $P_{450}$  catalyzed N-dealkylations reaction.

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### Highly active meso-microporous TaON photocatalyst driven by visible light

Seigo Ito, K. Ravindranathan Thampi,\* Pascal Comte, Paul Liska and Michael Grätzel

TaON was found to be ca. 20 times more active as a visible light activated photocatalyst for the oxidation of methanol, when compared to the well studied UV-visible activated  $TiO_2$  (P25) catalyst.



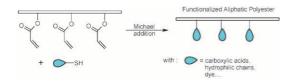
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## Sequential homobimetallic catalysis: an unprecedented tandem Pd(0)-catalysed deprotection – Pd(II)-catalysed heterocyclisation reaction leading to benzofurans

Bartolo Gabriele,\* Raffaella Mancuso, Giuseppe Salerno and Lucia Veltri

An unprecedented sequential Pd(0)-catalysed deallylation – Pd(II)-catalysed heterocyclisation reaction selectively leading to benzofuran-2-acetic esters in high yields has been developed.

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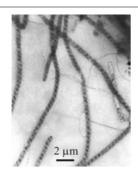
### Versatile functionalization and grafting of poly(ε-caprolactone) by Michael-type addition

J. Rieger, K. Van Butsele, P. Lecomte, C. Detrembleur, R. Jérôme and C. Jérôme\*

Michael-type addition is a versatile reaction for the anchoring of a large range of molecules to aliphatic polyesters, so providing them with new properties, including reactivity and amphiphilicity.

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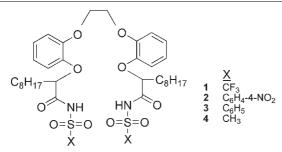


#### Formation of helical superstructures from a semifluorinated alkoxysilane through a surface and solution self-assembly process

Guanglei Cui, Hai Xu, Wei Xu,\* Guangcui Yuan, Deqing Zhang, Lei Jiang\* and Daoben Zhu\*

Helical superstructures were obtained through a surface and solution self-assembly process when a semi-fluorinated alkoxysilane modified silicon wafer was immersed in water of pH values ranging from 5.0 to 7.0 for more than one month.

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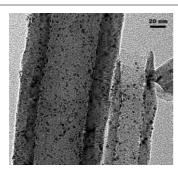


## Highly selective Ba<sup>2+</sup> separations with acyclic, lipophilic di-[*N*-(X)sulfonyl carbamoyl] polyethers

Sadik Elshani, Sangki Chun, Bijan Amiri-Eliasi and Richard A. Bartsch\*

Novel, di-ionizable, lipophilic, acyclic polyethers show high selectivity for Ba<sup>2+</sup> over other alkali metal cations in competitive metal ion separations.

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#### The two-step chemical vapor deposition of Pd(allyl)Cp as an atom-efficient route to synthesize highly dispersed palladium nanoparticles on carbon nanofibers

Changhai Liang, Wei Xia, Hamideh Soltani-Ahmadi, Oliver Schlüter, Roland A. Fischer and Martin Muhler\*

Highly dispersed palladium nanoparticles supported on carbon nanofibers, which show high catalytic activity and stability in the hydrogenation of cyclooctene, were synthesized by the two-step metal organic chemical vapor deposition of allylcyclopentadienylpalladium as precursor at atmospheric pressure.

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