

In this issue...

This review summarises the recently developed alternative enamine, enamide, and enol ether syntheses using cross-coupling reactions starting from vinyl halides and sulfonates. See Carsten Bolm et al. pp. 973-986.



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Cover

Structures of the half spin crossover (left) and high spin (right) forms of a doubly triazole-bridged diiron(II) complex superimposed on the Registry building of the University of Otago, New Zealand's first University. See p. 987. Image reproduced by permission of Marco H. Klingele, Boujemaa Moubaraki, John D. Cashion, Keith S. Murray and Sally Brooker.

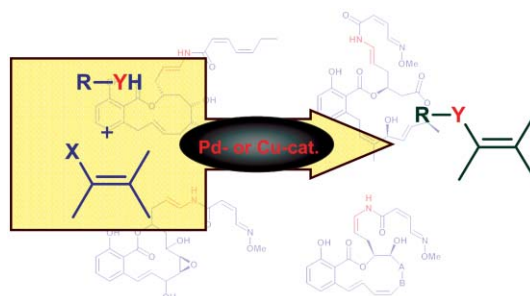
FEATURE ARTICLE

973

Synthesis of enamines, enol ethers and related compounds by cross-coupling reactions

Juan R. Dehli, Julien Legros and Carsten Bolm*

Palladium or copper catalysts promote the reactions between amines or alcohols and vinyl halides, leading to enamines and enol ethers. These reactions proceed under very mild reaction conditions and have already been applied in syntheses of natural products.



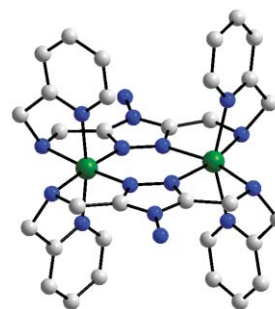
COMMUNICATIONS

987

The first X-ray crystal structure determination of a dinuclear complex trapped in the [low spin-high spin] state: $[\text{Fe}^{\text{II}}_2(\text{PMAT})_2](\text{BF}_4)_4 \cdot \text{DMF}$

Marco H. Klingele, Boujemaa Moubaraki, John D. Cashion, Keith S. Murray* and Sally Brooker*

The metal centres of the doubly 1,2,4-triazole-bridged dinuclear spin crossover complex $[\text{Fe}^{\text{II}}_2(\text{PMAT})_2](\text{BF}_4)_4 \cdot \text{DMF}$ are trapped in two different spin states below *ca.* 200 K, with no evidence that this particular [LS-HS] species can be converted into the [LS-LS] form at ambient pressure.



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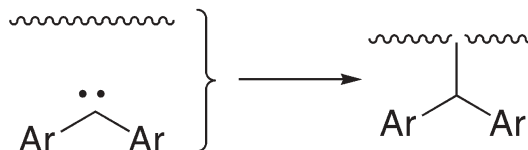
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990

A chemical method for the convenient surface functionalisation of polymers

Karim M. Awenat, Philip J. Davis, Mark G. Moloney* and Warren Ebenezer

The surface modification of several polymers using diaryl carbenes has been demonstrated, and the method has been used to generate coloured polymeric materials.

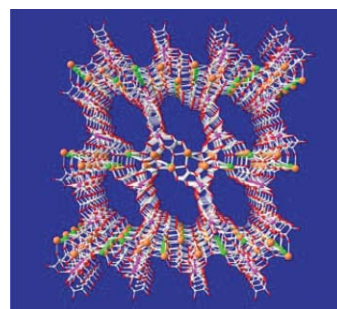


993

Structural and zeolitic features of a 3D heterometallic porous architecture constructed from a $\{M(\text{oxalate})_4\}^{4-}$ building unit

Inhar Imaz, Georges Bravic and Jean-Pascal Sutter*

The utilization of the tetrahedral pre-formed coordination compound $\{U(\text{C}_2\text{O}_4)_4\}^{4-}$ permitted the efficient synthesis of a 3D heteronuclear supramolecular nano-porous architecture undergoing a reversible sorption process without collapsing.

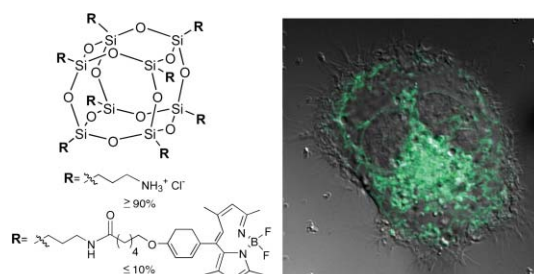


996

Cationic polyhedral oligomeric silsesquioxane (POSS) units as carriers for drug delivery processes

Catherine McCusker, Joseph B. Carroll and Vincent M. Rotello*

Quaternary ammonium functionalized polyhedral oligomeric silsesquioxane (OctaAmmonium-POSS[®]) units, widely employed as additives in ceramic and polymeric systems, possess many attributes which make them attractive as biocompatible drug carriers: nanoscale size, three-dimensional functionality, efficient cellular uptake, low toxicity, and high solubility.



999

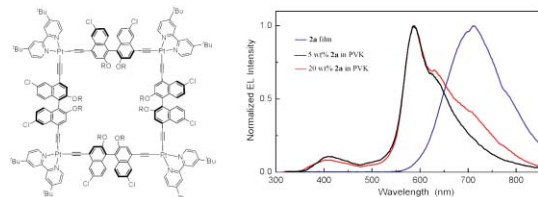
“Uncorking” of liposomes by matrix metalloproteinase-9

Nihar R. Sarkar, Theresa Rosendahl, Aaron B. Krueger, Abir L. Banerjee, Keith Benton, Sanku Mallik* and D. K. Srivastava*

A triggered release methodology of liposomal contents *via* the enzyme matrix metalloproteinase-9 is described.



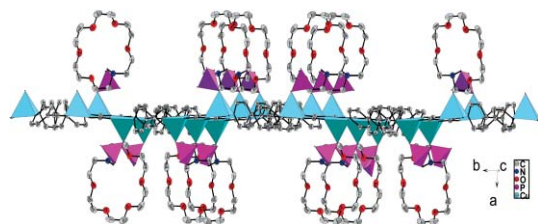
1002

**A highly electroluminescent molecular square**

Lin Zhang, Yu-Hua Niu, Alex K.-Y. Jen* and Wenbin Lin*

Chiral molecular triangles and squares containing the Pt(diiimine) metallocorners were prepared by a self-assembly process. The chiral molecular square exhibits efficient $^3\text{MLCT}$ emissions and is highly electroluminescent with a maximum brightness of 5470 cd m^{-2} and a maximum luminous efficiency of 0.93 cd A^{-1} .

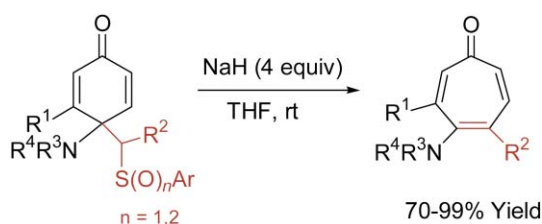
1005

**Novel copper macrocyclic leaflet with *N*-phosphonomethyl-monoaza-18-crown-6**

Deyuan Kong and Abraham Clearfield*

A novel 1D copper macrocyclic leaflet chain has been synthesized hydrothermally and its crystal structure determined.

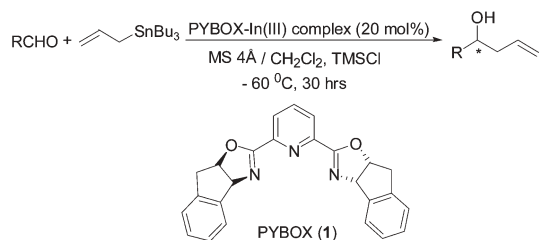
1007

**Ring expansion of sulfur substituted *p*-quinamines: regioselective synthesis of 4-aminotropones**

M. Carmen Carreño,* M. Jesús Sanz-Cuesta and María Ribagorda

Synthesis of 4-aminotropones through a cyclization–ring expansion process occurs in a single step and with excellent yields from 4-amino-2,5-cyclohexadienones (*p*-quinamines) bearing a 4-sulfinyl or sulfonyl methyl group.

1010

**Enantioselective synthesis of homoallylic alcohols *via* a chiral In(III)–PYBOX complex**

Jun Lu, Mei-Ling Hong, Shun-Jun Ji* and Teck-Peng Loh*

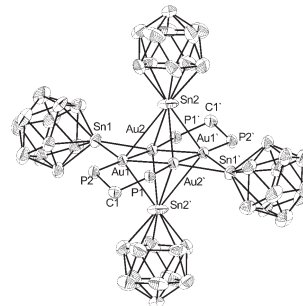
In the presence of 20 mol% of a chiral catalytic complex prepared from $\text{In}(\text{OTf})_3$ and chiral PYBOX, allyltributylstannane reacted with aldehydes to afford the corresponding homoallylic alcohols in moderate to high enantioselectivities (60–93% ee). This constitutes the first example of the enantioselective allylation of aldehydes catalyzed by a chiral $\text{In}(\text{III})$ –PYBOX complex.

1013

Formation of novel anionic gold–tin cluster compounds

Siegbert Hagen, Lars Wesemann* and Ingo Pantenburg

Reaction of the tin nucleophile $[\text{SnB}_{11}\text{H}_{11}]^{2-}$ with two gold complexes resulted in the formation of Au/Sn clusters with novel Sn_2Au_3 and Sn_2Au_4 cores.

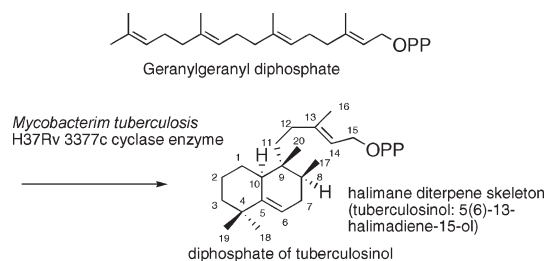


1016

Mycobacterium tuberculosis H37Rv3377c encodes the diterpene cyclase for producing the halimane skeleton

Chiaki Nakano, Tomoo Okamura, Tsutomu Sato, Tohru Dairi and Tsutomu Hoshino*

Mycobacterium tuberculosis H37Rv3377c encodes the diterpene cyclase to give a halimane skeleton, called tuberculosinol. It is of particular interest that this gene is involved only in the pathogenic species among mycobacteria.

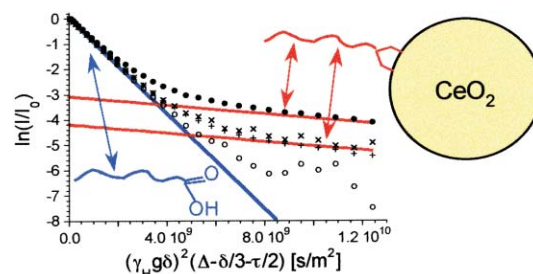


1019

In situ evaluation of interfacial affinity in CeO_2 based hybrid nanoparticles by pulsed field gradient NMR

François Ribot,* Virginie Escax, Claire Roiland, Clément Sanchez, José C. Martins, Monique Biesemans, Ingrid Verbruggen and Rudolph Willem

Complexation affinity of laurate ligands ($\text{C}_{12}\text{H}_{23}\text{O}_2$) grafted onto the surface of cerium(IV) oxide nanoparticles can be probed and quantified *in situ*, by pulsed field gradient ^1H NMR through the dependence of the diffusion coefficient on the size of a species.

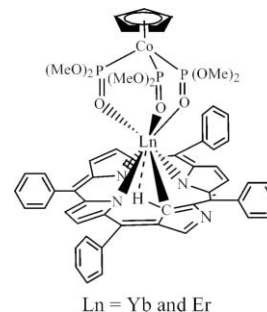


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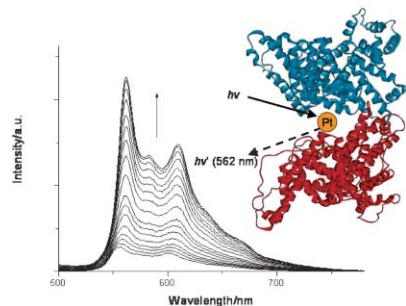
Synthesis and crystal structure of the first lanthanide complex of *N*-confused porphyrin with an η^2 agostic C–H interaction

Xunjin Zhu, Wai-Kwok Wong,* Wing-Kit Lo and Wai-Yeung Wong

The synthesis and characterization of the first lanthanide complex of *N*-confused porphyrin is described and the crystal structure shows the presence of an η^2 agostic C–H interaction.



1025

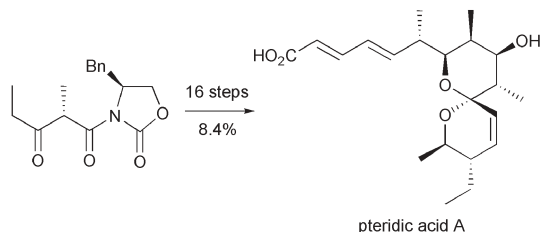


Luminescent cyclometalated platinum(II) complexes with amino acid ligands for protein binding

Phyllis Kit-Man Siu, Dik-Lung Ma and Chi-Ming Che*

The “Pt(C^N)(amino acid)” complexes have been found to bind to HSA [the human serum albumin (HSA) protein structure is generated from data obtained from a protein data bank and rendered by the WebLab ViewerLite 4.2 program], and the selectivity of such a binding reaction is dependent on the amino acid ligand and accompanied by photoluminescence enhancement.

1028

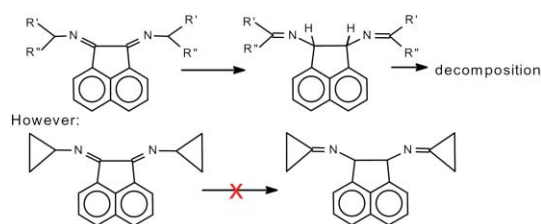


Enantioselective total synthesis of pteridic acid A

Takashi Nakahata and Shigefumi Kuwahara*

The first total synthesis of (+)-pteridic acid A, a potent plant growth promoter produced by *Streptomyces hygrosopicus* TP-A0451 isolated from a plant, was accomplished in 16 steps from a known oxazolidinone derivative by using the Evans asymmetric aldol reaction as the key C–C bond forming step.

1031



Using ring strain to inhibit a decomposition path: first synthesis of an Alkyl-BIAN ligand (Alkyl-BIAN = bis(alkyl)acenaphthenequinonediimine)

Fabio Ragaini,* Michela Gasperini, Emma Gallo and Piero Macchi

N-Alkyl imines of acenaphthenequinone are not stable because an isomerization occurs that releases part of the ring strain. This can be avoided by introducing an even more strained moiety in the alkylamine.

1034



A polymer support with controllable solubility in mutually immiscible solvents

Luca Minati and Andrea Biffis*

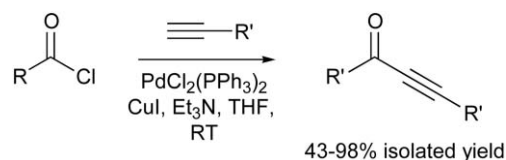
Complete control of the partition of cross-linked soluble polymer supports (microgels) in mutually immiscible solvents has been achieved, which can be exploited for the separation and purification of supported species (in this case, metal nanoclusters).

1037

Room temperature palladium catalysed coupling of acyl chlorides with terminal alkynes

Russell J. Cox,* Dougal J. Ritson, Thomas A. Dane, John Berge, Jonathan P. H. Charmant and Anob Kantacha

Conditions for the coupling of acyl chlorides with terminal acetylenes are reported. The reaction is tolerant of a wide range of functional groups, it is rapid under mild RT conditions and isolated yields are generally high.



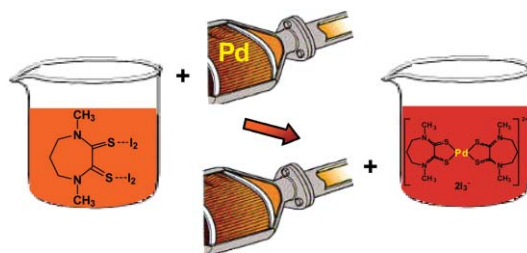
1040

Pd-Dissolution through a mild and effective one-step reaction and its application for Pd-recovery from spent catalytic converters

Angela Serpe, Francesco Bigoli, M. Cristina Cabras, Paolo Fornasiero, Mauro Graziani, M. Laura Mercuri, Tiziano Montini, Luca Pilia, Emanuele F. Trogu and Paola Deplano*

The powerful and safe oxidation reagent *N,N'*-dimethylperhydrodiazepine-2,3-dithione (Me_2dazdt) diiodine adduct is capable to dissolve Pd-metal forming $[\text{Pd}(\text{Me}_2\text{dazdt})_2]\text{I}_6$ in a mild one-step reaction and to recover palladium from model spent catalytic converters almost quantitatively.

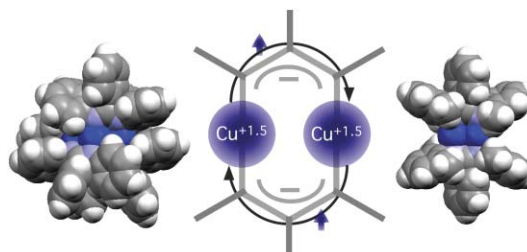
1043



Copper clusters built on bulky amidinate ligands: spin delocalization via superexchange rather than direct metal-metal bonding

Xuan Jiang, John C. Bollinger, Mu-Hyun Baik* and Dongwhan Lee*

Entry into a new class of copper clusters was assisted by fine steric tuning of bulky amidinate ligands, which provides spin-delocalizing superexchange pathways for class III dicopper(I,II) cores having short metal-metal distances.

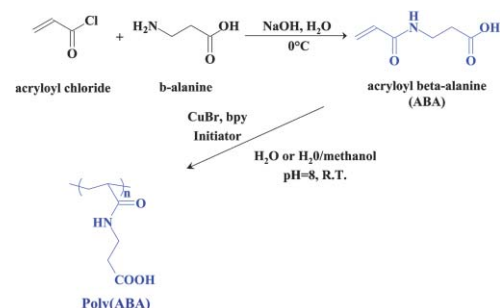


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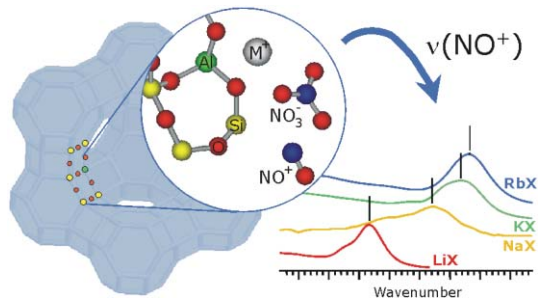
Synthesis of amino acid-based polymers via atom transfer radical polymerization in aqueous media at ambient temperature

Il-Doo Chung, Philip Britt, Dong Xie, Eva Harth and Jimmy Mays*

Well-defined acryloyl β -alanine (ABA) polymers were synthesized directly via atom transfer radical polymerization (ATRP) under near physiological conditions using various water soluble initiators with high yield and narrow molecular weight distributions.



1049

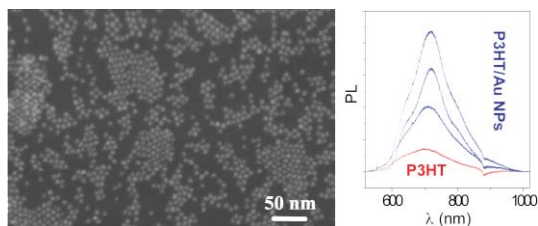


NO₂ disproportionation for the IR characterisation of basic zeolites

Olivier Marie, Nicolas Malicki, Catherine Pommier, Pascale Massiani, Ann Vos, Robert Schoonheydt, Paul Geerlings, Carlos Henriques and Frédéric Thibault-Starzyk*

Nitrosonium ions (NO⁺) on zeolites are infrared probes for cation chemical hardness and offer unprecedented efficiency for measuring the basicity of zeolitic oxygen atoms.

1052

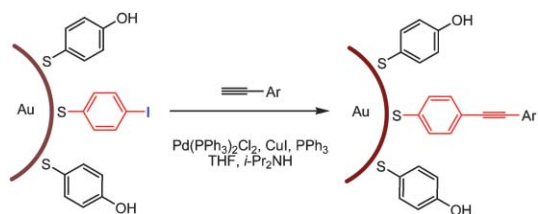


Enhanced visible photoluminescence in ultrathin poly(3-hexylthiophene) films by incorporation of Au nanoparticles

Patrick G. Nicholson, Virginia Ruiz, Julie V. Macpherson* and Patrick R. Unwin*

Incorporation of non-luminescent dodecanethiolate-protected gold clusters into regioregular poly(3-hexylthiophene) films results in a 6-fold increase in the visible photoluminescence (PL) of the polymer, which arises predominantly from nanoparticle (NP)-induced structural changes in the composite films.

1055

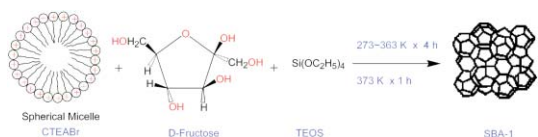


Construction of conjugated molecular structures on gold nanoparticles *via* the Sonogashira coupling reactions

Cuihua Xue, Ganesh Arumugam, Kumaranand Palaniappan, Stephen A. Hackney, Haiying Liu* and Jian Liu*

Functional, conjugated molecular structures have been fabricated on Au nanoparticles *via* the Sonogashira coupling reactions.

1058



Facile synthesis of stable cubic mesoporous silica SBA-1 over a broad temperature range with the aid of D-fructose

Hsien-Ming Kao,* Chun-Chiang Ting, Anthony S. T. Chiang, Chih-Chun Teng and Cheng-Ho Chen

The addition of D-fructose, combined with cetyltriethylammonium bromide (CTEABr) and tetraethylorthosilicate (TEOS) as the surfactant and silicon source, respectively, facilitates the synthesis of well-ordered SBA-1 over a broad temperature range.

1061

Revisiting the Maitland–Japp reaction. Concise construction of highly functionalised tetrahydropyran-4-ones

Paul A. Clarke,* William H. C. Martin, Jason M. Hargreaves, Claire Wilson and Alexander J. Blake

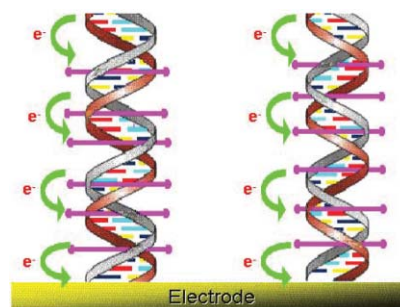
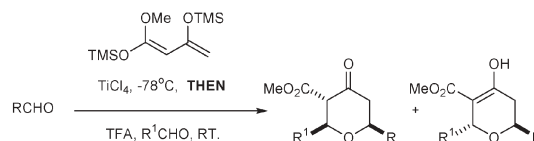
2005 is the 101st anniversary of the Maitland–Japp reaction! Three simple molecules can be coaxed to assemble into highly functionalised tetrahydropyran-4-ones by treatment with a Lewis acid. An aldol reaction is followed by a Knoevenagel condensation and a Michael reaction all in one pot. Enantioenriched aldol products can be used to form enantioenriched tetrahydropyran-4-ones with no loss of enantiomeric integrity.

1064

An ultrasensitive nucleic acid biosensor based on the catalytic oxidation of guanine by a novel redox threading intercalator

Natalia C. Tansil, Fang Xie, Hong Xie and Zhiqiang Gao*

An electrocatalytic intercalator, *N,N'*-bis[(3-propyl)imidazole]-1,4,5,8-naphthalene diimide imidazole complexed with Ru(bpy)₃Cl was synthesized in an attempt to develop a sensitive nucleic acid biosensor. An electrocatalytic signal was observed when as little as 7.5×10^{-18} moles of TP53 was present.

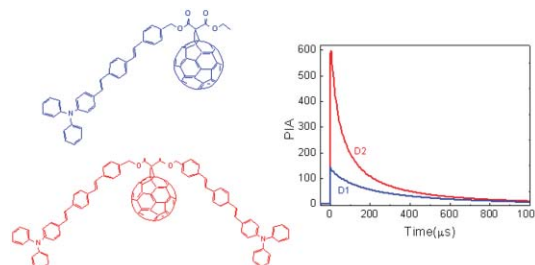


1067

Millisecond long-lived charge separated state at room temperature in a flexibly linked diphenylaminopolyene-C₆₀ dyad

Yanong Han, Laura Dobeck, Aijun Gong, Fanqing Meng, Charles W. Spangler and Lee H. Spangler*

A long-lived photoinduced charge separation is achieved in dyads coupling diphenylaminopolyene donors and C₆₀ with a flexible linkage. A new scheme allows simultaneous analysis of first and second order recombinations.

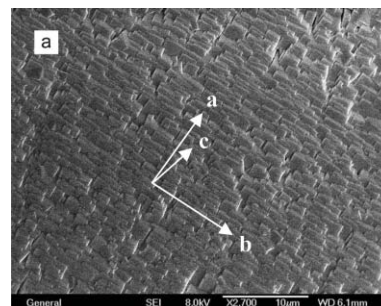


1070

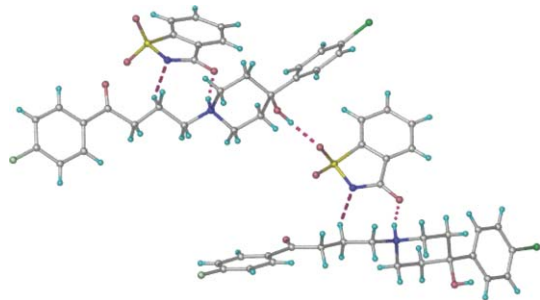
Preparation of an ordered zeolite MFI film by epitaxial growth

Jian Sun, Guangshan Zhu,* Xiaoju Yin, Yue Chen, Yanjie Cui and Shilun Qiu*

An ordered zeolite MFI film has been prepared on the {001} basal face of large silicalite-1 crystals by epitaxial growth. It provides more information for investigating the mechanism of “seeded growth”, an important method to prepare zeolite films.



1073

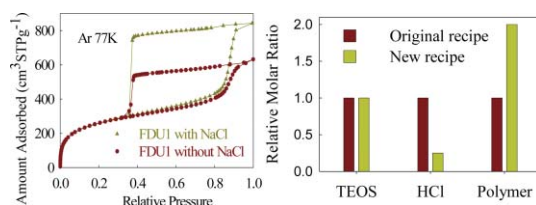


Saccharin as a salt former. Enhanced solubilities of saccharinates of active pharmaceutical ingredients

Prashant M. Bhatt, Nittala V. Ravindra, Rahul Banerjee and Gautam R. Desiraju*

Saccharin, acting as a weak acid, forms salts with basic APIs and these salts have the desirable property of enhanced water solubility. A spoonful of 'sugar' makes the medicine go down.

1076

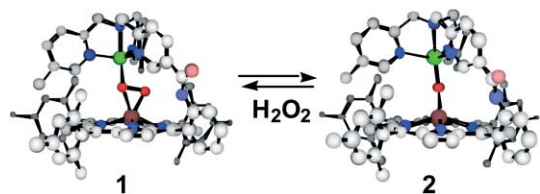


Cage-like ordered silica with large mesopore volume synthesized by doubling amount of polymer, adding sodium chloride and lowering acid concentration

Rafal M. Grudzien and Mietek Jaroniec*

Ordered mesoporous silica, FDU-1, with a large pore volume and narrow pore size distribution was synthesized by doubling the amount of block copolymer, adding sodium chloride and lowering the acid concentration.

1079

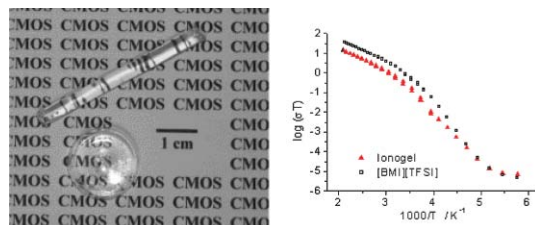


Selective formation of a stable μ-peroxy ferric heme-Cu^{II} complex from the corresponding μ-oxo Fe^{III}-Cu^{II} species with hydrogen peroxide

Takefumi Chishiro, Yuichi Shimazaki, Fumito Tani and Yoshinori Naruta*

An oxo-bridged heme-copper complex, [(TMP)Fe^{III}-(O)-(5MeTPA)Cu^{II}]BPh₄ (2), obtained by thermal transformation of the corresponding peroxo-bridged dinuclear complex, [(TMP)Fe^{III}-(O₂)-(5MeTPA)Cu^{II}]BPh₄ (1), was reacted with 1 equiv. H₂O₂ to regenerate 1 quantitatively by ligand exchange, without the formation of ferryl-oxo species or heme degradation.

1082



A route to heat resistant solid membranes with performances of liquid electrolytes

Marie-Alexandra Néouze, Jean Le Bideau, Fabrice Leroux and André Vioux*

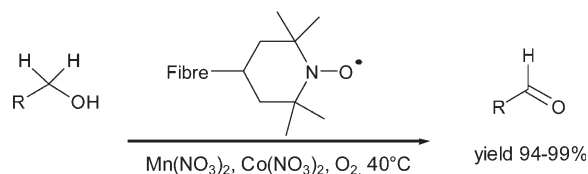
The one-pot sol-gel synthesis of silica-based solids containing nanoconfined ionic liquids is reported. These new materials are endowed with both the transparency and mechanical properties of silica and the conductivity of ionic liquids.

1085

Polymer-supported nitroxyl radical catalyst for selective aerobic oxidation of primary alcohols to aldehydes

Michelle Gilhespy, Martin Lok and Xavier Baucherel*

PS-TEMPO, a polymer-supported TEMPO, was successfully applied as a heterogeneous recyclable catalyst for the selective aerobic oxidation of primary alcohols to aldehydes in the presence of $\text{Mn}(\text{NO}_3)_2$ and $\text{Co}(\text{NO}_3)_2$.

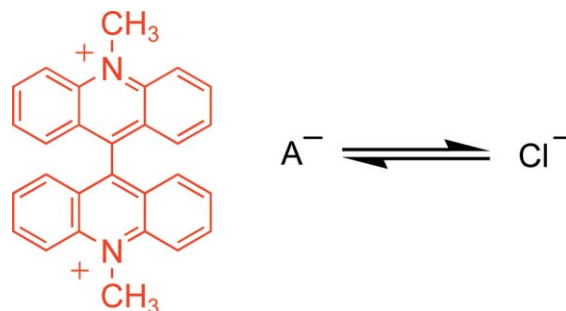


1087

A fluorescent assay for chloride transport; identification of a synthetic anionophore with improved activity

Beth A. McNally, Atanas V. Koulov, Bradley D. Smith,* Jean-Baptiste Joos and Anthony P. Davis*

A fluorescent assay based on the chloride-sensitive probe, lucigenin, is developed to monitor chloride transport into vesicles. The assay shows that a steroid-derived transport carrier with very high chloride affinity has excellent transport activity.

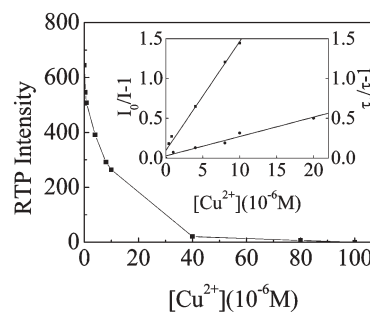


1090

Selective sensing of Cu(II) at ng ml^{-1} level based on phosphorescence quenching of 1-bromo-2-methylnaphthalene sandwiched in sodium deoxycholate dimer

Yu Wang, Jian-Jun Wu, Yu-Feng Wang, Li-Pin Qin and Wei-Jun Jin*

Using sodium deoxycholate as a protective medium, the selective recognition of $\text{Cu}(\text{II})$ at ng ml^{-1} level is realized through dynamic phosphorescence quenching of 1-bromo-2-methylnaphthalene (BMN) without deoxygenation. The limit of detection is 4.32 ng ml^{-1} , and the relative standard deviation is 1% at $10 \mu\text{M}$, linear up to $1 \times 10^{-5} \text{ M}$.

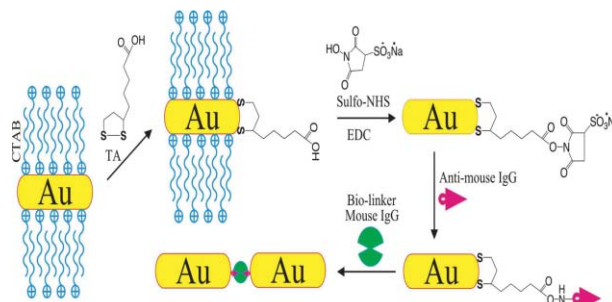


1092

Oriented assembly of Au nanorods using biorecognition system

Jia-Yaw Chang, Huimeng Wu, Hui Chen, Yong-Chien Ling and Weihong Tan*

Left: Schematic illustration of antibody-antigen directed assembly of gold nanorods. Gold nanorods modified with anti-mouse IgG can be assembled in the presence of mouse IgG to different nanostructures with desired orientations by controlling the concentrations of the biomolecules. Right: SEM pictures of assembled nanorodes.




AUTHOR INDEX

- Arumugam, Ganesh, 1055
 Awenat, Karim M., 990
 Baik, Mu-Hyun, 1043
 Banerjee, Abir L., 999
 Banerjee, Rahul, 1073
 Baucherel, Xavier, 1085
 Benton, Keith, 999
 Berge, John, 1037
 Bhatt, Prashant M., 1073
 Biesemans, Monique, 1019
 Biffis, Andrea, 1034
 Bigoli, Francesco, 1040
 Blake, Alexander J., 1061
 Bollinger, John C., 1043
 Bolm, Carsten, 973
 Bravic, Georges, 993
 Britt, Philip, 1046
 Brooker, Sally, 987
 Cabras, M. Cristina, 1040
 Carreño, M. Carmen, 1007
 Carroll, Joseph B., 996
 Cashion, John D., 987
 Chang, Jia-Yaw, 1092
 Charmant, Jonathan P. H., 1037
 Che, Chi-Ming, 1025
 Chen, Cheng-Ho, 1058
 Chen, Hui, 1092
 Chen, Yue, 1070
 Chiang, Anthony S. T., 1058
 Chishiro, Takefumi, 1079
 Chung, Il-Doo, 1046
 Clarke, Paul A., 1061
 Clearfield, Abraham, 1005
 Cox, Russell J., 1037
 Cui, Yanjie, 1070
 Dairi, Tohru, 1016
 Dane, Thomas A., 1037
 Davis, Anthony P., 1087
 Davis, Philip J., 990
 Dehli, Juan R., 973
 Deplano, Paola, 1040
 Desiraju, Gautam R., 1073
 Dobeck, Laura, 1067
 Ebenezer, Warren, 990
 Escax, Virginie, 1019
 Fornasiero, Paolo, 1040
 Gallo, Emma, 1031
 Gao, Zhiqiang, 1064
 Gasperini, Michela, 1031
 Geerlings, Paul, 1049
 Gillespy, Michelle, 1085
 Gong, Aijun, 1067
 Graziani, Mauro, 1040
 Grudzien, Rafal M., 1076
 Hackney, Stephen A., 1055
 Hagen, Siegbert, 1013
 Han, Yanong, 1067
 Hargreaves, Jason M., 1061
 Harth, Eva, 1046
 Henriques, Carlos, 1049
 Hong, Mei-Ling, 1010
 Hoshino, Tsutomu, 1016
 Imaz, Inhar, 993
 Jaroniec, Mietek, 1076
 Jen, Alex K.-Y., 1002
 Ji, Shun-Jun, 1010
 Jiang, Xuan, 1043
 Jin, Wei-Jun, 1090
 Joos, Jean-Baptiste, 1087
 Kantacha, Anob, 1037
 Kao, Hsien-Ming, 1058
 Kit-Man Siu, Phyllis, 1025
 Klingele, Marco H., 987
 Kong, Deyuan, 1005
 Koulov, Atanas V., 1087
 Krueger, Aaron B., 999
 Kuwahara, Shigefumi, 1028
 Le Bideau, Jean, 1082
 Lee, Dongwhan, 1043
 Legros, Julien, 973
 Leroux, Fabrice, 1082
 Lin, Wenbin, 1002
 Ling, Yong-Chien, 1092
 Liu, Haiying, 1055
 Liu, Jian, 1055
 Lo, Wing-Kit, 1022
 Loh, Teck-Peng, 1010
 Lok, Martin, 1085
 Lu, Jun, 1010
 Ma, Dik-Lung, 1025
 Macchi, Piero, 1031
 Macpherson, Julie V., 1052
 Malicki, Nicolas, 1049
 Mallik, Sanku, 999
 Marie, Olivier, 1049
 Martin, William H. C., 1061
 Martins, José C., 1019
 Massiani, Pascale, 1049
 Mays, Jimmy, 1046
 McCusker, Catherine, 996
 McNally, Beth A., 1087
 Meng, Fanqing, 1067
 Mercuri, M. Laura, 1040
 Minati, Luca, 1034
 Moloney, Mark G., 990
 Montini, Tiziano, 1040
 Moubaraki, Boujema, 987
 Murray, Keith S., 987
 Nakahata, Takashi, 1028
 Nakano, Chiaki, 1016
 Naruta, Yoshinori, 1079
 Néouze, Marie-Alexandra, 1082
 Nicholson, Patrick G., 1052
 Niu, Yu-Hua, 1002
 Okamura, Tomoo, 1016
 Palaniappan, Kumaranand, 1055
 Pantenburg, Ingo, 1013
 Pilia, Luca, 1040
 Pommier, Catherine, 1049
 Qin, Li-Pin, 1090
 Qiu, Shilun, 1070
 Ragaini, Fabio, 1031
 Ravindra, Nittala V., 1073
 Ribagorda, Maria, 1007
 Ribot, François, 1019
 Ritson, Dougal J., 1037
 Roiland, Claire, 1019
 Rosendahl, Theresa, 999
 Rotello, Vincent M., 996
 Ruiz, Virginia, 1052
 Sanchez, Clément, 1019
 Sanz-Cuesta, M. Jesús, 1007
 Sarkar, Nihar R., 999
 Sato, Tsutomu, 1016
 Schoonheydt, Robert, 1049
 Serpe, Angela, 1040
 Shimazaki, Yuichi, 1079
 Smith, Bradley D., 1087
 Spangler, Charles W., 1067
 Spangler, Lee H., 1067
 Srivastava, D. K., 999
 Sun, Jian, 1070
 Sutter, Jean-Pascal, 993
 Tan, Weihong, 1092
 Tani, Fumito, 1079
 Tansil, Natalia C., 1064
 Teng, Chih-Chun, 1058
 Thibault-Starzyk, Frédéric, 1049
 Ting, Chun-Chiang, 1058
 Trogu, Emanuele F., 1040
 Unwin, Patrick R., 1052
 Verbruggen, Ingrid, 1019
 Vioux, André, 1082
 Vos, Ann, 1049
 Wang, Yu, 1090
 Wang, Yu-Feng, 1090
 Wesemann, Lars, 1013
 Willem, Rudolph, 1019
 Wilson, Claire, 1061
 Wong, Wai-Kwok, 1022
 Wong, Wai-Yeung, 1022
 Wu, Huimeng, 1092
 Wu, Jian-Jun, 1090
 Xie, Dong, 1046
 Xie, Fang, 1064
 Xie, Hong, 1064
 Xue, Cuihua, 1055
 Yin, Xiaojun, 1070
 Zhang, Lin, 1002
 Zhu, Guangshan, 1070
 Zhu, Xunjin, 1022

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