# ChemComm

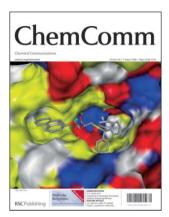
#### Chemical Communications

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#### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (29) 3029-3144 (2006)



#### Cover

See Kim D. Janda et al., page 3063. A perfect fit!! For the first time, a small molecule, non-peptidic, inhibitor of botulinum neurotoxin A protease displays neurotoxicity protection in a cellular model. The identification and characterization of this novel class of inhibitors is detailed. Image reproduced by permission of Grant E. Boldt, Lisa M. Eubanks and Kim D. Janda from Chem. Commun., 2006, 3063.

#### **FEATURE ARTICLE**

3045

#### Applications of chiral $C_3$ -symmetric molecules

Susan E. Gibson\* and M. Paola Castaldi

The undeniable beauty of  $C_3$ -symmetrical molecules has yet to be exploited in a major area of chemical endeavour. This review, which surveys the application of chiral  $C_3$ -symmetrical molecules in the areas of asymmetric catalysis, molecular recognition and nanostructures, aims to identify some of the most promising areas of application of these aesthetically appealing molecules.

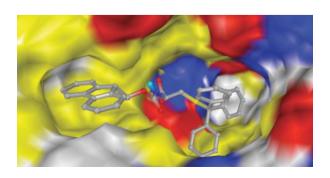
#### **COMMUNICATIONS**

3063

#### Identification of a botulinum neurotoxin A protease inhibitor displaying efficacy in a cellular model

Grant E. Boldt, Lisa M. Eubanks and Kim D. Janda\*

For the first time, a small-molecule, non-peptidic, inhibitor of botulinum neurotoxin A protease displays efficacy in a cellular model. The identification and characterization of this novel class of inhibitors are detailed.



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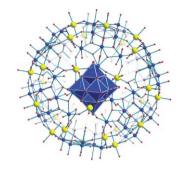
Formation of a "less stable" polyanion directed and protected by electrophilic internal surface functionalities of a capsule in growth:  $[\{Mo_6O_{19}\}^{2-} \subset \{Mo^{v_1}_{72}Fe^{III}_{30}O_{252}(ac)_{20}(H_2O)_{92}\}]^{4-}$ 

Achim Müller,\* Ana Maria Todea, Hartmut Bögge, Joris van Slageren, Martin Dressel, Anja Stammler and

Mariana Rusu

A deliberately constructed molybdenum oxide based capsule

A deliberately constructed molybdenum oxide based capsule shows concomitant with its growth the formation and final encapsulation of a hexamolybdate.



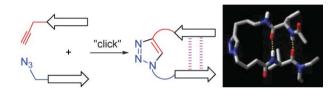
3069



## A convergent synthesis of new $\beta$ -turn mimics by click chemistry

Keunchan Oh and Zhibin Guan\*

A highly convergent strategy for constructing a new  $\beta$ -turn mimic is reported. Cycloaddition between peptide strands derivatized with azide and alkyne, respectively, provides an efficient synthesis of triazole ring-based  $\beta$ -turn mimics.



3072

## Linear oligomers composed of a photochromically contractible and extendable Janus [2]rotaxane

Susumu Tsuda, Yoshio Aso and Takahiro Kaneda\*

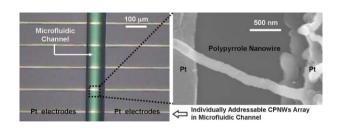
We report the first example of oligomeric molecular muscles, N,N'-p-xylylene-linked oligo-Janus [2]rotaxanes based on a permethylated  $\alpha$ -cyclodextrin—azobenzene hermaphrodite and their contractible and extendable nature coupled with photochromism.

3075

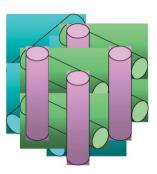
#### Electrochemical fabrication of conducting polymer nanowires in an integrated microfluidic system

Jun Wang, Yuri L. Bunimovich, Guodong Sui, Stavros Savvas, Jinyi Wang, Yaoyao Guo, James R. Heath\* and Hsian-Rong Tseng\*

A new approach for the *in situ* electrochemical fabrication of an individually addressable array of conducting polymer nanowires positioned within an integrated microfluidic device is introduced. The resulting integrated device can be used as a chemical sensor immediately after its construction.



4



## Orthogonal packing of enantiomerically pure helical silver coordination networks

Abdelaziz Jouaiti, Mir Wais Hosseini,\* Nathalie Kyritsakas, Phlippe Grosshans and Jean-Marc Planeix

The combination of an enantiopure tecton derived from the (R)-1,1'-binaphthyl backbone bearing two pyridine groups with a silver cation leads to the first example of enantiomerically pure helical strands with orthogonal packing in the rare space group  $I_2$ <sub>1</sub>3.

3081



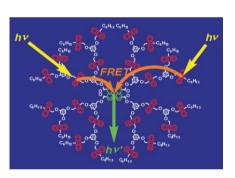
Design and synthesis of hydrophobic and chiral anions from amino acids as precursor for functional ionic liquids

Kenta Fukumoto and Hiroyuki Ohno\*

Hydrophobic ionic liquids composed of tetrabutylphosphonium cation and chiral anions derived from amino acids modified with trifluoromethanesulfonyl groups were synthesized with a simple method.

3084





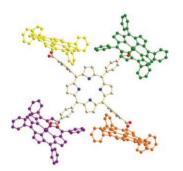
Evaluation of energy transfer in perylene-cored anthracene dendrimers

Masaki Takahashi,\* Hironao Morimoto, Kentaro Miyake, Mitsuji Yamashita, Hideki Kawai, Yoshihisa Sei and Kentaro Yamaguchi

Quantitative evaluation of Förster-type fluorescence resonance energy transfer (FRET) was undertaken by statistical investigations on perylene-cored anthracene dendrimers.

3087





Aluminium(III) porphyrins as supramolecular building blocks

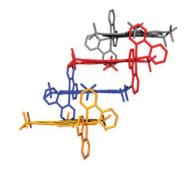
Gregory J. E. Davidson, Lok H. Tong, Paul R. Raithby and Jeremy K. M. Sanders\*

The ability of aluminium(III) porphyrins to coordinate strongly to one carboxylate ion and one nitrogenous ligand is exploited to generate new multiporphyrin arrays.

Intermolecular interactions of extended aromatic ligands: the synchrotron molecular structures of [Ru(bpy)<sub>2</sub>(N-HSB)].2PF<sub>6</sub> and [Ru(bpy)<sub>2</sub>(N- $\frac{1}{2}$ HSB)].2PF<sub>6</sub>

Daniel J. Gregg, Christopher M. Fitchett and Sylvia M. Draper\*

The planar N-HSB polyaromatic ligand in [Ru(bpy)<sub>2</sub>(N-HSB)].2PF<sub>6</sub> provides a platform for self-assembly into an off-set stack, whereas the distorted N-½HSB in [Ru(bpy)<sub>2</sub>(N-½HSB)].2PF<sub>6</sub> results in a saddle-like structure which cradles a PF<sub>6</sub> anion.

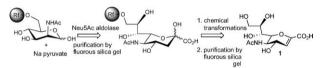


3093

#### Preparation of a fluorous protecting group and its application to the chemoenzymatic synthesis of sialidase inhibitor

Kiyoshi Ikeda,\* Hitomi Mori and Masayuki Sato\*

Neu5Ac aldolase-catalyzed chemoenzymatic transformation of N-acetyl-D-mannosamine to Neu5Ac derivatives was achieved successfully by using the fluorous reagent not only for hydroxy group protection but also for fluorous tagging. This chemoenzymatic method was applied to the synthesis of 2-deoxy-2,3-didehydrosialic acid 1 known as a potent sialidase inhibitor.



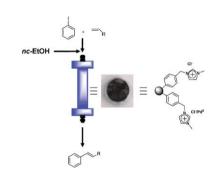
Rf = fluorous protecting group

3095

#### Pd(0) supported onto monolithic polymers containing IL-like moieties. Continuous flow catalysis for the Heck reaction in near-critical EtOH

Naima Karbass, Victor Sans, Eduardo Garcia-Verdugo,\* M. Isabel Burguete and Santiago V. Luis\*

Long-term stable Pd(0) catalysts can be easily supported onto polymeric monoliths containing methyl-imidazole moieties and the corresponding reactors based on these materials can be used for the continuous Heck reaction in near-critical EtOH.



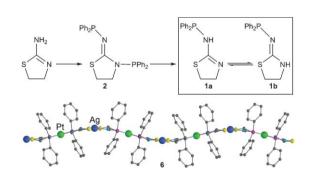
3098



#### Intra- and inter-molecular phosphoryl migration in phosphinothiazolines; precursors to polynuclear complexes and bimetallic coordination polymers

Günter Margraf, Roberto Pattacini, Abdelatif Messaoudi and Pierre Braunstein\*

Phosphinoaminothiazolines 1a,b and 2 were prepared and structurally characterized; their formation mechanism, elucidated by DFT calculations, features intra- and inter-molecular  $Ph_2P$  transfers. 1 was used for the formation of the Ag-Pt wave-like coordination polymer  $[Ag_{\infty}[Pt(1_{-H})_{2}]_{\infty}](OTf)_{\infty}$  (6).

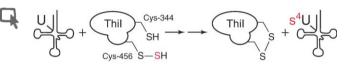


## Highly diastereoselective formation and reactions of a non-mesomerically stabilized, lithiated $\alpha$ -thiocarbanion

Ravindra P. Sonawane, Roland Fröhlich and Dieter Hoppe\*

Stereoselective generation of a new class of unbranched, non-mesomerically stabilized  $\alpha$ -thiocarbanion and its substitution with various electrophiles is described. The stereochemical course of the substitution was elucidated by NMR studies and X-ray crystal structure analysis.

3104



## Direct evidence for enzyme persulfide and disulfide intermediates during 4-thiouridine biosynthesis

Chapman M. Wright, Glenn D. Christman, Ann M. Snellinger, Murray V. Johnston and Eugene G. Mueller\*

Key intermediates in sulfur transfer have been definitively established in thionucleoside biosynthesis and are likely to be found in other sulfur transfer pathways that utilize sulfur mobilized by cysteine desulfurases.

3107

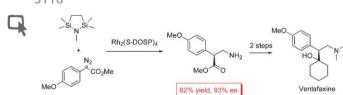


## Unusual mesomorphic behaviour of an ethynyl-substituted phthalocyanine

Eva M. García-Frutos, Giovanni Bottari, Purificación Vázquez,\* Joaquín Barberá\* and Tomás Torres\*

An ethynyl-substituted nickel(II) phthalocyanine has been synthesised and its thermotropic properties studied, revealing an unusual mesomorphic behaviour observed for the first time in phthalocyanine systems where each disk of the hexagonal columnar mesophase is formed by two ethynyl-substituted phthalocyanine units.

3110



# Enantioselective synthesis of $\beta$ -amino esters and its application to the synthesis of the enantiomers of the antidepressant Venlafaxine

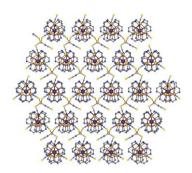
Huw M. L. Davies\* and Aiwu Ni

β-Amino esters are readily formed from the rhodium(II) prolinate-catalyzed intermolecular C–H insertion between methyl aryldiazoacetates and a bis-silyl protected methylamine. This chemistry was applied to a direct synthesis of Venlafaxine.

A new 2D network built from potassium sandwiches  $\{K[Cu^{II}{}_3(bdap)_3]_2\}$  and  $\{(\mu_{1,3}\text{-SCN})_3Cu^I(NCS)\}$  anions: structure and magnetic behaviour

M. Salah El Fallah,\* Fatima Badyine, Ramon Vicente, Albert Escuer, Xavier Solans and Mercè Font-Bardia

The synthesis of inorganic materials by using the dinuclear block [Cu<sub>2</sub>(bdap)]<sup>3+</sup> and alkali metal ions seems to be a good strategy to obtain new compounds with interesting molecular architectures and magnetic properties.

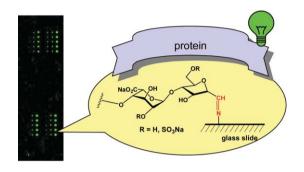


3116

Microarrays of heparin oligosaccharides obtained by nitrous acid depolymerization of isolated heparin

Jose L. de Paz, Dorothe Spillmann and Peter H. Seeberger\*

Heparin oligosaccharides derived by nitrous acid depolymerization of heparin have been immobilized on amine-coated glass slides. The formation of a Schiff base creates heparin chips that are a suitable platform for the highthroughput analysis of carbohydrate-protein interactions.



3119

A modular approach to the synthesis of 2,3,4-trisubstituted tetrahydrofurans

Christopher G. Nasveschuk, Nathan T. Jui and Tomislav Rovis

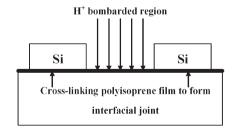
Three components are brought together in three steps to create three contiguous stereocenters about a THF core. The sequence is highlighted by a generally applicable and versatile Lewis acid mediated [1,3]-rearrangement of a vinyl acetal.

3122

A new cross-linking route via the unusual collision kinematics of hyperthermal protons in unsaturated hydrocarbons: the case of poly(trans-isoprene)

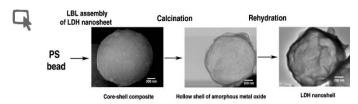
Zhi Zheng, Wai Man Kwok and Woon Ming Lau\*

The kinematics of 10 eV protons in unsaturated hydrocarbons like polyisoprene is harnessed to initiate cross-linked polymer formation with tailor-made chemistry even inside an interfacial enclosure or a micro/nano tube.



#### **COMMUNICATIONS**

3125

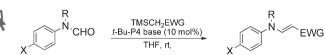


#### Hollow nanoshell of layered double hydroxide

Liang Li, Renzhi Ma, Nobuo Iyi, Yasuo Ebina, Kazunori Takada and Takayoshi Sasaki\*

Hollow nanoshells of layered double hydroxide (LDH) have been fabricated using exfoliated LDH nanosheets as a shell building block and polystyrene beads as a sacrificial template.

3128

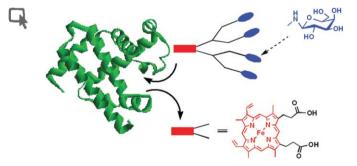


## Phosphazene base-catalyzed condensation of trimethylsilylacetate with carbonyl compounds

Koji Kobayashi, Masahiro Ueno and Yoshinori Kondo\*

The t-Bu-P4 base was found to be an excellent catalyst for the condensation of functionalized trimethylsilylalkanes with carbonyl compounds to form  $\beta$ -enaminoesters.

3131



## Construction of glycosylated myoglobin by reconstitutional method

Takashi Matsuo, Hirokazu Nagai, Yoshio Hisaeda and Takashi Hayashi\*

Artificial myoglobin with galactosyl interfaces was prepared by the reconstitutional method, in which the galactose units are attached at the terminal of the heme-propionate side chains through a flexible linker.

3134

## Monofluorinated di- and tetrahydropyrans via Prins-type cyclisations

Adrian P. Dobbs,\* Levan Pivnevi, Mark J. Penny, Săsa Martinović, James N. Iley and Peter T. Stephenson

The synthesis of a range of monofluorinated heterocycles is described via a Lewis acid-mediated Prins-type cyclisation and postulated to proceed via an  $\alpha$ -F carbocation.

#### **COMMUNICATIONS**

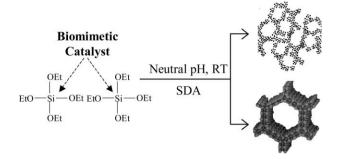
3137



Synthesis of micro- and mesoporous molecular sieves at room temperature and neutral pH catalyzed by functional analogues of silicatein

Avelino Corma,\* María J. Díaz-Cabañas, Manuel Moliner and Guillermo Rodríguez

By using functional mimics of the protein silicate  $\alpha$  together with organic structure directing agents, it was possible to produce different mesoporous and microporous molecular sieves at room temperature and neutral pH.



#### **ADDITION AND CORRECTION**

3140

Thermodynamic and kinetic factors in the hydrothermal synthesis of hybrid frameworks: zinc 4-cyclohexene-1,2dicarboxylates

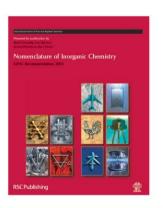
Clare Lee, Caroline Mellot-Draznieks, Ben Slater, G. Wu, William T. A. Harrison, C. N. R. Rao and Anthony K. Cheetham

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#### **AUTHOR INDEX**

Aso, Yoshio, 3072 Badyine, Fatima, 3113 Barberá, Joaquín, 3107 Bögge, Hartmut, 3066 Boldt, Grant E., 3063 Bottari, Giovanni, 3107 Braunstein, Pierre, 3098 Bunimovich, Yuri L., 3075 Burguete, M. Isabel, 3095 Castaldi, M. Paola, 3045 Christman, Glenn D., 3104 Corma, Avelino, 3137 Davidson, Gregory J. E., 3087 Davies, Huw M. L., 3110 de Paz, Jose L., 3116 Díaz-Cabañas, María J., 3137 Dobbs, Adrian P., 3134 Draper, Sylvia M., 3090 Dressel, Martin, 3066 Ebina, Yasuo, 3125 El Fallah, M. Salah, 3113 Escuer, Albert, 3113 Eubanks, Lisa M., 3063 Fitchett, Christopher M., 3090 Font-Bardia, Mercè, 3113 Fröhlich, Roland, 3101 Fukumoto, Kenta, 3081 García-Frutos, Eva M., 3107

Garcia-Verdugo, Eduardo, 3095 Gibson, Susan E., 3045 Gregg, Daniel J., 3090 Grosshans, Phlippe, 3078 Guan, Zhibin, 3069 Guo, Yaoyao, 3075 Hayashi, Takashi, 3131 Heath, James R., 3075 Hisaeda, Yoshio, 3131 Hoppe, Dieter, 3101 Hosseini, Mir Wais, 3078 Ikeda, Kiyoshi, 3093 Iley, James N., 3134 Iyi, Nobuo, 3125 Janda, Kim D., 3063 Johnston, Murray V., 3104 Jouaiti, Abdelaziz, 3078 Jui, Nathan T., 3119 Kaneda, Takahiro, 3072 Karbass, Naima, 3095 Kawai, Hideki, 3084 Kobayashi, Koji, 3128 Kondo, Yoshinori, 3128 Kwok, Wai Man, 3122 Kyritsakas, Nathalie, 3078 Lau, Woon Ming, 3122 Li, Liang, 3125 Luis, Santiago V., 3095

Ma, Renzhi, 3125 Margraf, Günter, 3098 Martinović, Săsa, 3134 Matsuo, Takashi, 3131 Messaoudi, Abdelatif, 3098 Miyake, Kentaro, 3084 Moliner, Manuel, 3137 Mori, Hitomi, 3093 Morimoto, Hironao, 3084 Mueller, Eugene G., 3104 Müller, Achim, 3066 Nagai, Hirokazu, 3131 Nasveschuk, Christopher G., 3119 Ni, Aiwu, 3110 Oh, Keunchan, 3069 Ohno, Hiroyuki, 3081 Pattacini, Roberto, 3098 Penny, Mark J., 3134 Pivnevi, Levan, 3134 Planeix, Jean-Marc, 3078 Raithby, Paul R., 3087 Rodríguez, Guillermo, 3137 Rovis, Tomislav, 3119 Rusu, Mariana, 3066 Sanders, Jeremy K. M., 3087 Sans, Victor, 3095 Sasaki, Takayoshi, 3125

Sato, Masayuki, 3093 Savvas, Stavros, 3075 Seeberger, Peter H., 3116 Sei, Yoshihisa, 3084 Snellinger, Ann M., 3104 Solans, Xavier, 3113 Sonawane, Ravindra P., 3101 Spillmann, Dorothe, 3116 Stammler, Anja, 3066 Stephenson, Peter T., 3134 Sui, Guodong, 3075 Takada, Kazunori, 3125 Takahashi, Masaki, 3084 Todea, Ana Maria, 3066 Tong, Lok H., 3087 Torres, Tomás, 3107 Tseng, Hsian-Rong, 3075 Tsuda, Susumu, 3072 Ueno, Masahiro, 3128 van Slageren, Joris, 3066 Vázquez, Purificación, 3107 Vicente, Ramon, 3113 Wang, Jinyi, 3075 Wang, Jun, 3075 Wright, Chapman M., 3104 Yamaguchi, Kentaro, 3084 Yamashita, Mitsuji, 3084 Zheng, Zhi, 3122

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