

# 803

## OCUS ARTICLE

## Bringing inorganic chemistry to life

## Achim Müller

The author describes his studies of the intricate and versatile chemical behaviour of molybdenum and its oxides, creating self-assembling systems of molybdates in solution that can be regarded as inorganic 'nanomodels' for biological activity at the cellular level.

### EATURE ARTICLE

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# Shape-persistent arylene ethynylene macrocycles: syntheses and supramolecular chemistry

Dahui Zhao and Jeffrey S. Moore\*

This article describes recent developments in the synthesis of macrocycles having rigid, monocyclic skeletons composed of arylene and ethynylene units and the studies on their self-assembling behavior.

### COMMUNICATIONS

Octanuclearity and tetradecanuclearity in manganese chemistry: an octanuclear manganese(II)/(III) complex featuring the novel  $[Mn_8(\mu_4-O)_2(\mu_3-OH)_2]^{14+}$  core and  $[Mn_{10}{}^{II}Mn_4{}^{III}O_4(O_2CMe)_{20}\{(2-py)_2C(OH)O\}_4]$  (2-py = 2-pyridyl)



Constantinos J. Milios, Elena Kefalloniti, Catherine P. Raptopoulou, Aris Terzis, Ramon Vicente, Nikolia Lalioti,\* Albert Escuer\* and Spyros P. Perlepes\*

The first members of the  $Mn_{10}^{II}Mn_4^{III}$  and  $Mn_4^{II}Mn_4^{III}$  sub-families of clusters are reported; tetradecanuclearity is extremely unusual in 3d-metal chemistry.

i.

# Chemical Communication

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Chemical Communications (print: ISSN 1359-7345; electronic: ISSN 1364-548X) is published 24 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF. All orders accompanied by payment should be sent directly to Turpin Distribution Services Ltd, Blackhorse Road, Letchworth, Herts, UK SG6 1HN, 2003 Annual (print + electronic) subscription price: £878; US\$1450. 2003 Annual (electronic) subscription price: £790; US\$1305. Customers in Canada will be subject to a surcharge to cover GST. Customers in the EU subscribing to the electronic version only will be charged VAT. If you take an institutional subscription to any RSC journal you are entitled to free, site-wide web access to that journal. You can arrange access via Internet Protocol (IP) address at www.rsc.org/ip. Customers should make payments by cheque in sterling payable on a UK clearing bank or in US dollars payable on a US clearing bank. Periodicals postage paid at Rahway, NJ, USA and at additional mailing offices. Airfreight and mailing in the USA by Mercury Airfreight International Ltd., 365 Blair Road, Avenel, NJ 07001, USA. US Postmaster: send address changes to Chemical Communications, c/o Mercury Airfreight International Ltd., 365 Blair Road, Avenel, NJ 07001. All despatches outside the UK by Consolidated Airfreight. PRINTED IN THE LIK

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Andrew B. Holmes, Cambridge, UK E-mail: abh1@cam.ac.uk

Frank Allen, CCDC, Cambridge, UK E-mail: allen@ccdc.cam.ac.uk Jerry L. Atwood, Columbia, MO, USA E-mail: rsc.chemcomm@missouri.edu Shankar Balasubramanian, Cambridge, UK F-mail: sb10031@cam ac uk Makoto Fujita, Tokyo, Japan E-mail: mfujita@appchem.t.u-tokyo.ac.jp Alois Fürstner, Mülheim, Germany E-mail: fuerstner@mpi-muelheim.mpg.de Donald Hilvert, Zurich, Switzerland E-mail: hilvert@org.chem.ethz.ch Wolfgang Hölderich, Aachen, Germany E-mail: Hoelderich@rwth-aachen.de

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### **Professor Dermot O'Hare**

Inorganic Chemistry Laboratory University of Oxford Oxford, UK E-mail: chemcomm@chem.ox.ac.uk

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SUPRAMOLECULAR **Professor Jerry L. Atwood** 123 Chemistry Building University of Missouri Columbia, MO, USA E-mail: rsc.chemcomm@missouri.edu

CHEMICAL BIOLOGY **Professor Barbara Imperiali** Department of Chemistry Massachusetts Institute of Technology Cambridge, MA, USA E-mail: chemcomm@mit.edu

Takuzo Aida, Tokyo, Japan Dario Braga, Bologna, Italy Duncan W. Bruce, Exeter, UK Jillian M. Buriak, West Lafayette, IN, USA David H. G. Crout, Warwick, UK Marcetta Darensbourg, College Station, TX, USA Gautam R. Desiraju, Hyderabad, India Pierre H. Dixneuf, Rennes, France Gregory C. Fu, Cambridge, MA, USA Tohru Fukuyama, Tokyo, Japan Lutz Gade, Strasbourg, France George W. Gokel, St Louis, MO, USA Karl J. Hale, London, UK Amir Hoveyda, Boston, MA, USA

**Professor Donald Hilvert** Laboratory of Organic Chemistry ETH Zentrum, Zurich, Switzerland E-mail: hilvert@org.chem.ethz.ch **Professor Mir Wais Hosseini** Lab de Chimie de Coordination Organique Universite Louis Pasteur, Strasbourg, France

> **Professor Alois Fürstner** Max-Planck-Institut für Kohlenforschung Müllheim/Ruhr, Germany

E-mail: fuerstner@mpi-muelheim.mpg.de

# INORGANIC, ORGANOMETALLIC AND MATERIALS

**Professor Ian Rothwell** Department of Chemistry Purdue University, West Lafayette, IN, USA E-mail: chemcomm@purdue.edu

ORGANIC **Professor James D. White** Department of Chemistry **Oregon State University** Corvallis, OR, USA

**Dr Sarah Thomas** Chemical Communications Royal Society of Chemistry Thomas Graham House Science Park, Milton Road Cambridge, UK. CB4 0WF Tel (+44) (0) 1223 420066 Fax (+44) (0) 1223 420247 E-mail: chemcomm@rsc.org

Kazuyuki Kuroda, Tokyo, Japan Jérôme Lacour, Geneva, Switzerland E. W. 'Bert' Meijer, Eindhoven, The Netherlands Albert I. Meyers, Fort Collins, CO, USA Jason Micklefield, Manchester, UK Achim Müller, Bielefeld, Germany Maurizio Prato, Trieste, Italy Richard J. Puddephatt, London, ON, Canada Christopher A. Reed, Riverside, CA, USA Jonathan Sessler, Austin, TX, USA David C. Sherrington, Glasgow, UK Jonathan W. Steed, London, UK Herbert Waldmann, Dortmund, Germany Henry N. C. Wong, Hong Kong, PR China

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Mir Wais Hosseini, Strasbourg, France E-mail: hosseini@chimie.u-strasbg.fr Barbara Imperiali, Cambridge, MA, USA E-mail: chemcomm@mit.edu Roeland J. M. Nolte, Nijmegen, The Netherlands E-mail: nolte@sci.kun.nl Dermot O'Hare, Oxford, UK E-mail: chemcomm@chem.ox.ac.uk Colin Raston, Perth, Australia E-mail: clraston@chem.uwa.edu.au David Rice, Reading, UK E-mail: c.foote@reading.ac.uk Ian Rothwell, West Lafayette, IN, USA E-mail: chemcomm@purdue.edu Clément Sanchez, Paris, France E-mail: clems@ccr.jussieu.fr James D. White, Corvallis, OR, USA E-mail: james.white@orst.edu

E-mail: hosseini@chimie.u-strasbg.fr

E-mail: james.white@orst.edu



Marina Lovrinovic, Ralf Seidel, Ron Wacker, Hendrik Schroeder, Oliver Seitz, Martin Engelhard, Roger S. Goody and Christof M. Niemeyer\*

The rapid and controlled covalent coupling of nucleic acids and bioactive proteins is achieved by expressed protein ligation. The resulting conjugates are versatile molecular tools for biochip technologies and nano sciences.

# Isolation of the first ferromagnetically coupled Mn(III/IV) complex

T. M. Rajendiran, Martin L. Kirk,\* Ika A. Setyawati, M. Tyler Caudle, Jeff W. Kampf and Vincent L. Pecoraro\*

The imidazolate bridge in the Mn<sub>2</sub>(III/IV)(dtsalpn)<sub>2</sub>DCBI induces a ferromagnetic exchange interaction between manganese ions resulting in an  $S_T = 7/2$  ground state.

# The structure of a self-assembled calixarene aqua-channel system

Anthony W. Coleman,\* Eric Da Silva, Farid Nouar, Martine Nierlich and Alda Navaza\*

The structure of a self-assembled calix-[4]-arene in the presence of propane diamine, water and ethanol into a dimeric structure of hexagonal units, each containing twelve calixarenes, containing an aqueous channel.

# Adsorbed water for the electro-oxidation of methanol at Pt-Ru alloy

Takahiro Yajima, Noriaki Wakabayashi, Hiroyuki Uchida and Masahiro Watanabe\*

Adsorbed water molecules which promote the methanol oxidation reaction at Pt-Ru alloy electrode are clearly detected for the first time by in-situ FTIR spectroscopy, which directly supports the "bi-functional mechanism".

# A 3D metal-organic network, [Cu<sub>2</sub>(glutarate)<sub>2</sub>(4,4'-bipyridine)], that exhibits single-crystal to single-crystal dehydration and rehydration

Beth Rather and Michael J. Zaworotko\*

Cu(glutarate)<sub>2</sub> forms 2D sheets that can be cross-linked by 4,4'-bipyridine and 1,2bis(4-pyridyl)ethane, thereby forming open framework 3D networks 1 and 2, respectively. Compound 1 (illustrated alongside) exhibits reversible desorption and adsorption of water molecules with retention of single crystallinity.

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# A reductive recycle strategy for the facile synthesis of molybdenum(VI) alkylidyne catalysts for alkyne metathesis

Wei Zhang, Stefan Kraft and Jeffrey S. Moore\*



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834

838

A convenient synthesis of trisamido molybdenum(VI) alkylidyne complexes has been developed, in which the key step is the addition of a geminal dichloride to a trisamido molybdenum(III) complex in the presence of magnesium to continuously recycle unwanted side product monochloride **4**, selectively generating the desired alkylidyne complexes in high yield.

### The role of the counteranion in the cation- $\pi$ interaction

Christopher A. Hunter,\* Caroline M. R. Low, Carmen Rotger, Jeremy G. Vinter and Cristiano Zonta

Chemical double mutant cycles have been used to quantify cation- $\pi$  interactions in chloroform as a function of the nature of the counteranion. The cation- $\pi$  interaction is  $-2.5 \pm 0.4$  kJ mol<sup>-1</sup> and independent of the anion, even though the overall stability of the complexes varies by an order of magnitude due to competition of the anion for alternative binding sites.



# ZnS-Zn nanocables and ZnS nanotubes

Ying-Chun Zhu,\* Yoshio Bando and Yoichiro Uemura

ZnS–Zn nanocables and ZnS nanotubes have been synthesized by a thermochemical process in a simple and safe way. The as-prepared nanocables consist of a single crystal Zn core with a diameter of 20 nm and a polycrystalline ZnS sheath with a thickness of 8 nm. The evaporation of the Zn core leads to the formation of ZnS nanotubes.

# Total synthesis of $(\pm)$ -dihydrospiniferin-1 *via* a polyfluoro alkanosulfonyl fluoride induced tandem carbonium ion rearrangement reaction

Ling Chen, Kai Ding and Wei-Sheng Tian\*



A novel polyfluoroalkanosulfonyl fluoride induced carbonium ion rearrangement reaction of  $\gamma$ -hydroxymethyl cyclohexenone has been used for the total synthesis of (±)dihydrospiniferin-1.

840

**fragments: synthesis of unprecedented** (η<sup>6</sup>-indene)ruthenium(II) **metallacycles** Victorio Cadierno, Salvador Coneiero, Josefina Díez, M. Pilar Gamasa, Jo



Victorio Cadierno, Salvador Conejero, Josefina Díez, M. Pilar Gamasa, José Gimeno\* and Santiago García-Granda

Unexpected coupling between an  $\eta^5$ -indenyl ligand and alkenyl-vinylidene

Vinylidene complexes [Ru{=C=C(H)CR<sup>1</sup>R<sup>2</sup>CH<sub>2</sub>C(Me)=CH<sub>2</sub>}( $\eta^{5}$ -C<sub>9</sub>H<sub>7</sub>)(PPh<sub>3</sub>)<sub>2</sub>][BF<sub>4</sub>] undergo an intramolecular coupling process between the alkenyl-vinylidene fragment and the  $\eta^{5}$ -indenyl ligand to afford unprecedented metallacyclic compounds in which the resulting functionalised indene unit is  $\eta^{6}$ coordinated to the metal.

iv





2

(3D)

1

2D)

# Palladium-catalysed dimerization of vinylarenes using indium triflate as an effective co-catalyst

Teruhisa Tsuchimoto, Susumu Kamiyama, Ryoju Negoro, Eiji Shirakawa\* and Yusuke Kawakami

Indium triflate as a co-catalyst drastically enhanced the reaction rate of the palladium-catalysed dimerization of vinylarenes by activating vinylarenes to add oxidatively to palladium(0) complexes.

# **RPM-2:** A recyclable porous material with unusual adsorption capability: self assembly via structural transformations

Long Pan, Haiming Liu, Sean P. Kelly, Xiaoying Huang, David H. Olson and Jing Li\*

Structural transformation via deliberate and partial topological changes in a grid network structure has led to a three-dimensional, fully recyclable porous material ( $\mathbb{R}PM-2$ ) with a very high sorption capability.

# $\begin{bmatrix} R^{3} R^{4} CO \\ R^{2} \\ P^{2} \\ P$

3

(1D)

854

856

# Dilithiated phosphazenes: scaffolds for the synthesis of olefins through a new class of bicyclic 1,2-oxaphosphetanes

Jesús García-López, Emma Peralta-Pérez, Angela Forcén-Acebal, Santiago García-Granda and Fernando López-Ortiz\*

Double lithiation at the  $C_{\alpha}$  and  $C_{ortho}$  to the phosphorus of (*N*-methoxycarbonyl)phosphazenes is reported. The dianions add to aldehydes and ketones affording a new type of isolable spirocyclic 1,2-oxaphosphetanes, that give rise to tri- and tetra-substituted olefins upon heating.



# Accelerated Bergman cyclization of porphyrinic-enediynes

Mahendra Nath, John C. Huffman and Jeffrey M. Zaleski\*

The Bergman cyclization of simple diethynylporphyrinic-enediynes exhibits a double activation barrier to the formation of Bergman cyclized product. Addition of H-atom acceptor accelerates the formation of the picenoporphyrin, indicating that the second barrier is rate limiting.



# Synthesis of transparent and ordered mesoporous silica monolithic films embedded with monomeric zinc phthalocyanine dye

Selvaraj Subbiah and Robert Mokaya\*

Nanocomposite monolithic films of zinc phthalocyanine–mesoporous silica may be prepared in which the embedded phthalocyanine exists predominantly in monomeric form.

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# Microporous niobium phosphates and catalytic properties prepared by a supramolecular templating mechanism

Nawal Kishor Mal, Asim Bhaumik, Prashant Kumar and Masahiro Fujiwara\*

Microporous distorted hexagonal niobium phosphate synthesized by a supramolecular templating mechanism ( $S^{0}I^{0}$ ) possesses strong hydrophilic character, which leads to high selectivity for catechol formation (95.3%) in the presence of protic solvent (MeOH) in the hydroxylation of phenol.

# Hydrogen-bonding based multilayer assemblies by self-deposition of dendrimer



Me

876

878

ОН

о́н

880

Fengwei Huo, Huaping Xu, Li Zhang, Yu Fu, Zhiqiang Wang and Xi Zhang\*

We reported on hydrogen-bonding directed Layer-by-Layer assemblies by self-deposition of a kind of dendrimer bearing carboxyl groups on its periphery that act as hydrogen bonding donor as well as hydrogen bonding acceptor.

# An unprecedented $\alpha$ -C–C agostic interaction in a cyclopropyl tris(pyrazolyl)boratoniobium complex

Joëlle Jaffart, Michel Etienne,\* Meike Reinhold, John E. McGrady\* and Feliu Maseras\*

A rare C–C agostic interaction is preferred over both  $\alpha$ - or  $\beta$ -C–H agostic alternatives in the cyclopropyl complex Tp<sup>Me2</sup>NbCl(c-C<sub>3</sub>H<sub>5</sub>)(MeCCMe). Calculations performed with the hybrid DFT/molecular mechanics methodology reveals that the origin of this preference is electronic rather than steric.

# High performance carbon-supported catalysts for fuel cells via phosphonation

Zhiqiang Xu, Zhigang Qi\* and Arthur Kaufman

Phosphonic acid groups have been successfully linked onto carbon-supported catalysts to effectively enhance the latter's performance in proton-exchange membrane fuel cells.

# A novel imidazolate-bridged heterodinuclear Cu(II)Zn(II) complex derived from a unique macrocyclic ligand with two hydroxyethyl pendants



Shu-an Li, Dong-feng Li,\* De-xi Yang, Yi-zhi Li, Jin Huang, Kai-bei Yu and (the late) Wen-xia Tang\*

A new imidazolate-bridged homodinuclear copper(II) complex **1** and a novel imidazolate-bridged heterodinuclear Cu(II)–Zn(II) complex **2**, derived from a single macrocyclic ligand with two flexible hydroxyethyl pendants, have been synthesized and characterized.

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H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>PO<sub>2</sub>F

Isobutyl nitrite

# 

884

888

# Synthesis and structure of a new layered zinc phosphite $(C_5H_6N_2)Zn(HPO_3)$ containing helical chains

Jing Liang, Yu Wang, Jihong Yu,\* Yi Li and Ruren Xu\*

A new compound  $(C_5H_6N_2)Zn(HPO_3)$  has been prepared hydrothermally; it consists of left-handed and right-handed helical chains that are connected through oxygen atoms to form an undulated sheet structure with 4.8-net.

# Solid state NMR study of acid sites formed by adsorption of $SO_3$ onto $\gamma\text{-}Al_2O_3$

Jun Yang, Mingjin Zhang, Feng Deng,\* Qing Luo, Delian Yi and Chaohui Ye

Detailed structure of Brønsted acid sites on the surface of  $SO_3/Al_2O_3$  catalyst has been proposed based on  ${}^{1}H/{}^{27}Al$  TRAPDOR NMR results and the acidity of the catalyst has also been characterized by NMR probe molecules.

# Ab initio structure study from in-house powder diffraction of a novel $ZnS(EN)_{0.5}$ structure with layered wurtzite ZnS fragment

Xiang Ouyang, Tsung-Yen Tsai, Dong-Hwang Chen, Qi-Jie Huang, Wu-Hsun Cheng and Abraham Clearfield

The solvothermal reaction of elemental zinc with sulfur in ethylenediamine (en) as solvent yields  $[ZnS \cdot 0.5(NH_2CH_2CH_2NH_2)]$ , **1**, an unprecedented ethylenediamine pillared ZnS layered compound, containing two dimensional (2-D) boat-type 6-membered rings, which was characterized by *ab initio* structure solution from powder diffraction data (SDPD).

# Self-assembly of a ferrocene-substituted porphyrin capable of electrochemically sensing neutral molecules *via* a "tail on-tail off" process

Christophe Bucher,\* Charles H. Devillers, Jean-Claude Moutet,\* Guy Royal and Eric Saint-Aman

Self-assembly of a novel functionalized ferrocene–porphyrin conjugate, with efficient electronic communication throughout the molecular receptor, allows unprecedented ferrocene-based electrochemical sensing of neutral species *via* a metalloporphyrin-centred "tail on–tail off" binding process.

# 8-(1,4,7,10-Tetraoxa-13-azacyclopentadec-13-ylmethyl)quinolin-7ol: synthesis and application as a highly sensitive metal cation probe

Kun-Chan Wu, Moawia O. Ahmed, Chun-Yan Chen, Guo-Wei Huang, Yung-Son Hon\* and Pi-Tai Chou\*

**1a** was proven to recognize metal cations incorporating excited-state proton transfer reaction. The remarkable differentiation in absorption and fluorescence makes **1a** a highly sensitive probe.













Takako Nakamura,\* Masatou Ishihara, Tsuguyori Ohana and Yoshinori Koga

Photolysis of perfluoroazooctane with diamond powders led to chemical modification of the surface by the introduction of perfluorooctyl ester and ether functional groups, the presence of which was confirmed by means of FT-IR, XPS and <sup>19</sup>F NMR measurements.

OC<sub>8</sub>F<sub>17</sub>

diamond

 $2 C_8 F_{17} + N_2$ 

CO₂H

CO2C8F17

diamond

diamond

C<sub>8</sub>F<sub>17</sub>N=NC<sub>8</sub>F<sub>17</sub>



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Reversible regulation of pyrene excimer emission by light and metal ions in the presence of photochromic spiropyran: toward creation of a new molecular logic circuit



A new fluorophore-photochrome system with the excimer

fluorescence as the output signal; reversible regulation of pyrene excimer fluorescence by light and metal ions in the presence of spiropyran.

Xuefeng Guo, Deqing Zhang,\* Tongxin Wang and Daoben Zhu\*

# Highly efficient Lewis acid-catalysed Pictet–Spengler reactions discovered by parallel screening

Natarajan Srinivasan and A. Ganesan\*

High yielding Lewis acid catalysed one-pot Pictet–Spengler reactions of tryptophan methyl ester and tryptamine with aliphatic and aromatic aldehydes were achieved in short reaction times with the aid of microwave irradiation.

## $R^1 = H \text{ or } CO_2 Me$

 $R^2$  = Phenyl, *p*-NO<sub>2</sub>-Phenyl, *p*-OMe-Phenyl, 3,4,5-(OMe)<sub>3</sub>-phenyl and *c*-C<sub>6</sub>H<sub>11</sub>

μω, 100 -120°C, 0.5

R<sup>2</sup>CHO / Lewis acid

- 1.0 h

## ADDITIONS AND CORRECTIONS

Valérie Atlan, Hugues Bienaymé, Laurent El Kaim and Adinath Majee The use of hydrazones for efficient Mannich type coupling with aldehydes and secondary amines

# )

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Kazuo Shinozuka, Noritake Matsumoto, Hideo Suzuki, Tomohisa Moriguchi and Hiroaki Sawai Alternate stranded triplex formation of chimeric DNA composed of tandem  $\alpha$ - and  $\beta$ -anomeric strands

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Dates, venues and contact details of forthcoming events.

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